

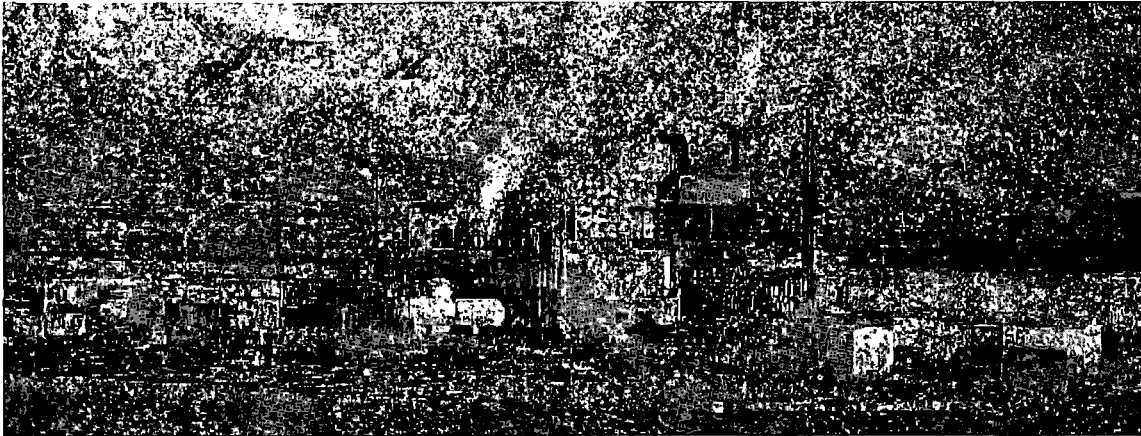
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JAN 07 2010

UTAH DIVISION OF
SOLID & HAZARDOUS WASTE

Tooele Chemical Agent Disposal Facility (TOCDF)



Request for a CLASS 3 MODIFICATION to the TOCDF RCRA Permit

Request Number	TOCDF-A10-03-1092
Request Title	Install and Operate Area 10 Incinerator
EPA ID Number	UT 5210090002

Attachment 3

**Final Report for Ton Container Sample
Analysis, October 2, 2009 and Addendum of
November 17, 2009**

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UTAH DIVISION OF
SOLID & HAZARDOUS WASTE
2010,00067

Final Report
For
Ton Container Sample Analysis

October 2, 2009

Prepared By
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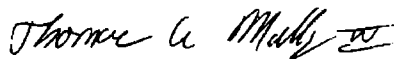
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Ton Container Sample Analysis

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Ton Container Sample Analysis

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A	Sample Information
B	Sampling Program Plan
C	Test Plan and Test Plan Change Memos

ACRONYMS

ACL	analytical chemistry laboratory
AMC	Army Materiel Command
BCO	Battelle Columbus Operations
BFB	4-bromofluorbenzene
CA	chemical agent
CAMDS	Chemical Agent Munitions Disposal System
CAS	Chemical Abstract Society
CB	chlorobenzene
CCV	continuing calibration verification
CEMO	Chemical, Environmental and Materials Operations
CoC	chain of custody
CMA	Chemical Materials Agency
CSM	chemical surety material
CVAA	2-chlorovinyl arsonous acid
CW	chemical warfare
DCD	Deseret Chemical Depot
DI	de-ionized
DFTPP	decafluorotriphenylphosphine
ECBC	Edgewood Chemical and Biological Center
EPA	Environmental Protection Agency
EQL	estimated quantitation limit
GA	tabun
GC	gas chromatography (<i>chromatograph</i>)
GC/FID	gas chromatography / flame ionization detection
GC/MS	gas chromatograph / mass spectrometer
HAZOP	hazardous operations
HML	hazardous materials laboratory
HMRC	Battelle's Hazardous Materials Research Center
HRA	Health Risk Assessment
HTM	highly toxic material
ICP-MS	inductively coupled mass spectrometry
IDL	instrument detection limit
IS	internal standard
L	Lewisite
LCS	laboratory control sample
L1	2-Chlorovinylarsonous dichloride
L2	Bis(2-chlorovinyl)arsinous chloride
L3	Tris(2-chlorovinyl)arsine
LITF	large item test facility
LRB	laboratory record book
MDL	method detection limit
MS	mass spectrometer, matrix spike
MSD	mass selective detector, matrix spike duplicate
NA	not applicable
ND	not determined
NIST	National Institute of Standards
OPCW	Organization for the Prevention of Chemical Weapons

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OSHA	Occupational Safety and Health Administration
PI	Principal Investigator
PFTBA	perfluorotributylamine
PM	Program Manager
PPE	personal protective equipment
PQL	practical quantitation limit
QA	quality assurance
QC	quality control
RDTE	research, development, testing and evaluation
RPD	relative percent difference
RSD	relative standard deviation
SRC	stock record card
SOP	standard operating procedure
TC	ton container
TIC	tentatively identified compounds
TMP	2,2,4-trimethylpentane
TOCDF	Toole Chemical Agent Destruction Facility
TPCS	test performance control sheet
Tr	calorimetry reactor temperature
VOC	volatile organic compound

1 0 INTRODUCTION

1 1 Background

Deseret Chemical Depot (DCD) currently stores ten ton containers (TCs) of Lewisite (L) comprising approximately 13 tons of agent and four ton containers of GA. Additionally, there exists ten TCs of what is described as 'transparency' TCs. The latter are believed to have contained Lewisite at one time, and have since been decontaminated and are considered empty. There are conflicting data, both documented and anecdotal, concerning the actual contents of these TCs. EG&G, DMI has been tasked by the Chemical Materials Agency (CMA) to develop means to destroy the agent and decontaminate the drained and transparency TCs for final disposal. EG&G has proposed incinerating the Lewisite from the TCs. The existing baseline incinerator systems would be unable to destroy the Lewisite in a timely and cost effective method, therefore EG&G has proposed modifying an existing small incinerator system built to destroy stocks of sulfur mustard. The incinerator system will be modified to remove the arsenic, mercury, and other metals that are prevalent in either the chemical structure of Lewisite or as a contaminant from the previous fill of the TCs.

The GA/Lewisite Sampling Program collected representative samples from the GA, GA/UCON, Lewisite and "transparency" TCs to support the processing in a liquid incinerator system to be constructed in Area 10 of the DCD. These samples were supplied to Battelle's Hazardous Materials Research Center (HMRC) for preparation and analysis. The results of the analysis will allow engineering plans and controls to be added to the planned disposal system to aid the incineration of the agent and the clearing of the TCs for off-site disposal.

The drained Lewisite TC will also need to be treated in order to meet the conditions of the Organization for the Prevention of Chemical Weapons (OPCW) treaty and also to meet the U.S. Army's conditions for off-site commercial disposal. EG&G originally proposed to meet these treatment conditions using a series of rinses. The drained Lewisite TCs will be rinsed with 20% acetic acid solution that will act as an organic solvent and a carrier for the remaining Lewisite to be destroyed in the incinerator. One or more rinses with the acetic acid may be necessary. Following the acetic acid rinse(s), the TC will be rinsed one or more times with a 7.0 M nitric acid solution. The nitric acid will dissolve any remaining metals in the liquid, oxidize any Lewisite, and remove the embedded metals in the pores of the TC.

1 2 Objectives

To sufficiently characterize GA and Lewisite agent feed to the liquid incinerator in a manner that is acceptable to the State of Utah, Division of Solid and Hazardous Waste. Characterization tests will include

- agent purity,
- tentatively identified compounds (TIC),
- density,
- pH,
- chlorobenzene content,
- L1, L2 and L3 content,
- Health Risk Assessment (HRA) metals and
- volatile organic compounds (VOC)

The objective of the rinse testing is to be able to use the test results to support the proposed rinsing of the ton containers

1 3 Limitations

Because stock Lewistite, containing primarily L1, was used to prepare instrument calibration standards only L1 was quantitatively determined in samples. The presence of L2 and L3 were qualitatively determined.

Only a single sorbent tube headspace sample was collected from each "transparency" ton container, therefore, re-analysis of these samples was not possible as the entire sample was consumed during the thermal desorption process.

1 4 Sample Information

See Appendix A for copies of DD 1911 forms, chains of custody, sampling data and HMRC stock record cards.

2 0 PROCEDURES AND RESULTS

2 1 Overview

The first part of this testing involved characterizing liquid and sludge samples collected from four GA ton containers and ten Lewisite ton containers. Air samples collected from ten "transparency" ton containers were also analyzed. The second part of this testing involved evaluating a procedure designed to mimic the proposed rinsing of residual Lewisite from drained ton containers. This testing also involved collecting calorimetric data collected from mixing Lewisite with acetic acid and with nitric acid.

Eight different tests were performed to characterize agent samples, as shown in Table 1. The following types and numbers of samples were received:

- GA liquid – 4 samples plus 1 field duplicate
- Lewisite liquid – 10 samples plus 1 field duplicate
- Lewisite sludge – 10 samples
- Transparency ton vapor – 10 samples

Holding time for sample analysis, per US Environmental Protection Agency (EPA) guidelines, was typically 60 days, except for Hg and VOC analysis, which was 28 day, all other metals had a 6-month hold time. Not all samples were analyzed by all tests. See Section 2.2 for a description of tests. See Appendix B for the Sampling Program Plan and a description of analytical method Quality Control criteria.

Table 1 GA and L Characterization Sample Matrix

Prep & Analysis	Method	GA Liquid	L Liquid	L Sludge	Transparency Ton Vapor
pH	9045D	5	11	-	-
Density	Volume and Mass	5	11	-	-
Purity	HMRC IV-055	5	11	-	-
Tentatively Identified Compounds (TICs)	HMRC IV-055	5	11	-	-
Chlorobenzene	HMRC IV-056	5	-	-	-
VOCs	HMRC IV-067	-	-	-	10
HRA Metals	HMRC X-241	5	11	10	-

2 2 Sample Descriptions

Descriptions of samples are shown in Table 2. Representative photographs of each sample type are shown in Figure 1. Sample D79685-S-01 appeared to have a greater liquid content (liquidy) than the sludges that are described as "wet".

Table 2 Description of Agent Samples

Sample ID Number	SRC Number	Agent	Color	Consistency	Number of Phases
D-49221-S-01	W006	Lewisite (sludge)	Black	Dry Tar	1
D-79685-S-01	W009	Lewisite (sludge)	Black	Liquidy Tar	1
D-79693-S-01	W012	Lewisite (sludge)	Black	Dry Tar	1
D-79697-S-01	W014	Lewisite (sludge)	Black	Dry Tar	1
D-79699-S-01	W016	Lewisite (sludge)	Black	Dry Tar	1
D-79700-S-01	W018	Lewisite (sludge)	Black	Wet Tar	1
D-79701-S-01	W020	Lewisite (sludge)	Black	Dry Tar	1
D-79703-S-01	W022	Lewisite (sludge)	Black	Wet Tar	1
D-79705-S-01	W024	Lewisite (sludge)	Black	Dry Tar	1
D-79711-S-01	W026	Lewisite (sludge)	Black	Wet Tar	1
D-25253-L-01	W001	GA	Brown	Viscous Liquid	1
D-25253-L-D	W002	GA	Brown	Viscous Liquid	1
D-35248-L-01	W003	GA	Brown	Viscous Liquid	1
D-29813-L-01	W004	GA	Brown	Viscous Liquid	1
D-51365-L-01	W005	GA	Brown	Viscous Liquid	1
D-49221-L-01	W007	Lewisite	Black	Liquid	1
D-49221-L-D	W008	Lewisite	Black	Liquid	1
D-79685-L-01	W010	Lewisite	Black	Liquid	1
D-79693-L-01	W013	Lewisite	Black	Liquid	1
D-79697-L-01	W015	Lewisite	Black	Liquid	1
D-79699-L-01	W017	Lewisite	Black	Liquid	1
D-79700-L-01	W019	Lewisite	Black	Liquid	1
D-79701-L-01	W021	Lewisite	Black	Liquid	1
D-79703-L-01	W023	Lewisite	Black	Liquid	1
D-79705-L-01	W025	Lewisite	Black	Liquid	1
D-79711-L-01	W027	Lewisite	Black	Liquid	1

SRC = stock record card number assigned by the HMRC



Figure 1. Representative photos of Lewisite sludge D-79703-S-01 (left), GA liquid D-25253-L-01 (middle) and Lewisite liquid D-79697-L-01 (right).

2.3 GA and L Characterization

2.3.1 pH for GA and Lewisite liquid samples

Measurement of pH for GA and Lewisite liquid samples was to be performed by EPA Method 9045D using a pH meter. A 1-g sample was going to be used instead of the 20-g sample specified by the method. However, the use of this method with GA was not possible. When 1.1 g of GA, from sample D-25253-L01, was mixed with 1.1 mL of deionized (DI) water and allowed to sit, no visible phase separation occurred. This was likely due to the chemical compound that gave the GA its brown coloration, dissolving in the DI water turning it brown as well. This chemical compound was demonstrated to be polar during GA preparation for purity analysis, as it would not dissolve in hexane (it precipitated on the bottom of the vial) but did dissolve in methylene chloride. As it was not possible to insert the pH electrode into the water phase, since it could not be discerned, the pH determination for all GA samples was measured by placing the pH electrode directly into the neat GA. All pH measurements for GA and Lewisite were made using a Jenco Model 6320N pH meter with a Pinnacle series pH electrode (catalog #476436). Both electrodes used for testing were calibrated following the pH meter manufacturer's guidelines.

Additionally, based on information reviewed regarding the hydrolysis of Lewisite, it was determined that the use of EPA Method 9045D was not appropriate for this agent. According to Munro et al.¹, hydrolysis is rapid, resulting in the formation of the water-soluble 2-chlorovinyl arsonous acid (CVAA). Also, according to Haigh², 1 mole of Lewisite undergoes a fast reaction with 2 mole of water to form 1 mole of CVAA and 2 mole of HCl; this hydrolysis reaction is quantitative. While no half-life or kinetic data are available in the literature, the above information indicated that the use of Method 9045D, with the 1:1 mixing of sample and water, was not appropriate – particularly since the formation of HCl will result in pH measurement that is biased low. Additionally, a rapid hydrolysis reaction may have resulted in the evolution of significant amounts of heat and therefore presented a safety concern. Lewisite sample pH was instead measured by placing the pH electrode directly into the neat Lewisite.

Table 3 summarizes the pH results for GA and Lewisite. The pH for each of the five GA TC samples was measured in replicate using pH electrode 1 (serial # C082414079). The electrode tip was rinsed with DI water following each measurement. After the second measurement, the electrode response was checked using pH 7 calibration standard, for GA the calibration check passed acceptance criteria. As can be seen from the results, the first GA measurement was higher than the second measurement. This may have resulted from an influence of the previous pH reading on the actual sample measurement. For the first pH measurement the previous pH reading was from the pH 7 calibration check solution, for the second pH measurement, the previous pH reading was from the DI water rinse, which was slightly more acidic than the pH 7 calibration check solution. As direct pH measurement of organic liquids is heavily dependent on water content of the organic liquid, it can be difficult to obtain an accurate pH reading. The measurements obtained here for GA can be considered a good representation of the likely pH range for these samples.

The pH for Lewisite was only measured for two ton container samples. When pH electrode 1 was placed in sample D-49221-L-01, the pH measurement rapidly dropped to a final reading of -1.10, indicating a mechanical failure of the electrode. A replicate measurement for this sample was not taken. When the pH electrode was placed in pH 7 calibration solution, the measured pH was 3.24, also indicating probe failure. A second electrode (serial # C082414076) was used to take a pH measurement of a second sample (D-79685-L-01) with similar results. The final pH measurement was -1.36. Again, the pH 7 calibration check failed with a measurement of 2.74. As these results indicated that the pH of Lewisite could not be accurately measured and that taking the measurements damaged the pH electrodes, pH testing for Lewisite was terminated.

Table 3 GA and L pH

Sample ID Number	SRC Number	Agent	pH Electrode ID	Replicate 1 pH	Replicate 2 pH	pH 7 Cal. Check
D-25253-L-01	W001-1-1	GA	1	5.28	3.24	7.06
D-25253-L-D	W002-1-1	GA	1	6.16	3.04	7.04
D-35248-L-01	W003-1-1A	GA	1	6.39	4.87	6.99
D-29813-L-01	W004-1-1	GA	1	7.70	4.25	6.93
D-51365-L-01	W005-1-1	GA	1	7.78	4.09	6.98
D-49221-L-01	W007	L	1	-1.10	ND	3.24*
D-79685-L-01	W010	L	2	-1.36	ND	2.74*

* Outside of acceptance range SRC = stock record card ND = not determined

2.3.2 Density for GA and Lewisite liquid samples

A 250 µL Class A syringe was used to deposit 50.0 µL of agent onto a balance and the mass recorded to 0.1 mg. The balance was calibrated with National Institute of Standards (NIST) traceable weights daily prior to use, including a sensitivity check. Measurements were performed at ambient temperature. Each sample was weighed in duplicate along with one preparation blank using de-ionized water. Density was calculated as measured mass divided by measured volume. Sample measurements and calculated densities are shown in Table 4. The average density for the five GA samples was 1.13 g/mL and the average density for the eleven Lewisite samples was 2.00 g/mL. Relative percent difference was calculated as the absolute difference between replicate densities divided by the replicate density average. No RPD was greater than 2.2%, within the 10% quality control (QC) acceptance limit.

Table 4 GA and L Density

Sample ID Number	SRC Number	Agent	Rep 1 Vol (μL)	Rep 1 Wt (g)	Rep. 1 Density (g/mL)	Rep 2 Vol (μL)	Rep 2 Wt (g)	Rep 2 Density (g/mL)	Rep. 1 & 2 Avg. (g/mL)	Rep. 1 & 2 RPD
Preparation Blank	NA	NA	50 0	0 0503	1 01	50 0	0 0505	1 01	1 01	0 4%
D-25253-L-01	W001-1-1	GA	50 0	0 0553	1 11	50 0	0 0549	1 10	1 10	0 7%
D-25253-L-D	W002-1-1	GA	50 0	0 0572	1 14	50 0	0 0585	1 17	1 16	2 2%
D-35248-L-01	W003-1-1A	GA	50 0	0 0573	1 15	50 0	0 0567	1 13	1 14	1 1%
D-29813-L-01	W004-1-1	GA	50 0	0 0534	1 07	50 0	0 0544	1 09	1 08	1 9%
D-51365-L-01	W005-1-1	GA	50 0	0 0584	1 17	50 0	0 0582	1 16	1 17	0 3%
D-49221-L-01	W007	L	50 0	0 1006	2 01	50 0	0 0985	1 97	1 99	2 1%
D-49221-L-D	W008	L	50 0	0 0999	2 00	50 0	0 1014	2 03	2 01	1 5%
D-79685-L-01	W010	L	50 0	0 0973	1 95	50 0	0 0978	1 96	1 95	0 5%
D-79693-L-01	W013	L	50 0	0 1012	2 02	50 0	0 1007	2 01	2 02	0 5%
D-79697-L-01	W015	L	50 0	0 1008	2 02	50 0	0 1004	2 01	2 01	0 4%
D-79699-L-01	W017	L	50 0	0 1004	2 01	50 0	0 1000	2 00	2 00	0 4%
D-79700-L-01	W019	L	50 0	0 0997	1 99	50 0	0 0995	1 99	1 99	0 2%
D-79701-L-01	W021	L	50 0	0 0989	1 98	50 0	0 0997	1 99	1 99	0 8%
D-79703-L-01	W023	L	50 0	0 1007	2 01	50 0	0 1001	2 00	2 01	0 6%
D-79705-L-01	W025	L	50 0	0 1008	2 02	50 0	0 0992	1 98	2 00	1 6%
D-79711-L-01	W027	L	50 0	0 1017	2 03	50 0	0 1012	2 02	2 03	0 5%

SRC = stock record card NA = not applicable RPD = relative percent difference

2 3 3 Chlorobenzene for GA liquid samples

Samples were prepared by diluting 10 μ L of neat agent in 10 mL of methylene chloride and analyzed by GC/MS following guidelines of SOP HMRC-IV-056. A five-point calibration curve was prepared in methylene chloride ranging from 10 μ g/mL to 200 μ g/mL. 1,4-dichlorobenzene- d_4 was added as an internal standard to all calibration standards and samples at a concentration of 20 μ g/mL. Prior to instrument calibration, a GC/MS tune check was performed using (decafluorotriphenylphosphine) DFTPP. Sample results are shown in Table 5. Chlorobenzene content ranged from 4.1 to 13 weight % (40,800 to 122,000 mg/Kg). All QC samples met acceptance criteria. RPD values (determined from mg/Kg concentrations) for the field duplicate and batch duplicate were 0.2% and 1.5%, respectively, indicating excellent sample collection and analytical method reproducibility.

Table 5 Chlorobenzene in GA

Sample Name	Data File Name	CB Conc (mg/mL)*	GA Density (g/mL)	CB Conc (mg/Kg)	CB (Weight %)	CB Recovery	RPD
Prep Blank	09030912.D	<10					
LCS	09030922.D	12.5				125%	
D-25253-L-01	09030913.D	44.9	1.10	40,800	4.1%		0.2%
D-25253-L-D	09030914.D	47.4	1.16	40,900	4.1%		
D-35248-L-01	09030915.D	148	1.14	130,000	13.0%		1.5%
D-35248-L-01 DUP	09030916.D	151	1.14	132,000	13.2%		
D-29813-L-01	09030918.D	125	1.08	116,000	11.6%		
D-29813-L-01 MS	09030919.D	132	1.08	122,000	12.2%	67%	20%
D-29813-L-01 MSD	09030920.D	131	1.08	121,000	12.1%	55%	
D-51365-L-01	09030921.D	125	1.17	107,000	10.7%		

* mg of chlorobenzene per mL of sample

CB = chlorobenzene LCS = laboratory control sample

RPD = relative percent difference

2 3 4 Agent purity and TICs for GA liquid samples

Purity samples were prepared per standard operating procedure (SOP) HMRC IV-055 and quantitatively analyzed per SOP HMRC IV-056. Samples were prepared by diluting 10 μ L of neat agent in 10 mL of methylene chloride. A five-point calibration curve for GA, ranging from 25.5 μ g/mL to 255 μ g/mL, was also prepared in methylene chloride. Standards and samples were analyzed by gas chromatography / flame ionization detection (GC/FID) using a splitless injection. Weight percent purity was calculated by converting the measured concentration of GA (mg/mL) in the prepared samples to a mass / mass (mg/Kg) concentration using the measured density. Sample results are shown in Table 6.

GA purity ranged from 19.8 % to 39.7%. These low purities are consistent with the large number of other components detected in the TIC analysis (see below). The RPD value for the field duplicate was 4.6%, indicating good sample collection reproducibility. The RPD value for the batch duplicate was 1.9%, indicating very good analytical method reproducibility.

Table 6 GA Purity

Sample Name	GA Conc (mg/mL)*	GA Density (g/mL)	GA Conc (mg/Kg)	GA (Weight %)	RPD
Prep Blank	<25.5				
D-25253-L-01	437	1.10	397,000	39.7%	4.6%
D-25253-L-D	439	1.16	379,000	37.9%	
D-35248-L-01	299	1.14	263,000	26.3%	1.9%
D-35248-L-01 DUP	306	1.14	268,000	26.8%	
D-29813-L-01	228	1.08	211,000	21.1%	
D-51365-L-01	232	1.17	198,000	19.8%	

* mg of GA per mL of sample

RPD = relative percent difference

Tentatively identified compounds were determined following SOP HMRC IV-055. The data files from the sample analysis for chlorobenzene were reviewed and the peaks with a response of at least 1% of the internal standard were selected. The mass spectra for each peak was compared to spectra from a NIST 2002 mass spectral library for tentative identification. Spectra with a quality match (Q) ≥ 75 were reported as the library identified compound. All other compounds were reported as belonging to a chemical class or as unknown. See Table 7 for a list of tentatively identified compounds. Because an internal standard (IS) was added to these samples, a semi-quantitative TIC concentration could be calculated based on the TIC area response, the known IS concentration and the measured IS area response. These TIC concentrations, in units of mg of detected TIC per mL of GA, are estimates only and should be treated as such. The estimated concentrations are reported to one significant figure. Several compounds were found in all samples. Many compounds could not be identified based on comparison of the compound's mass spectrum to library spectra. If a TIC was not detected in a sample then no concentration estimate was provided in Table 7. Also, Chemical Abstract Society (CAS) numbers could not be provided for unknown compounds. Note that the estimated concentration for GA is significantly higher than what was determined through quantitative analysis.

The analysis of samples for TICs, when performed with the addition of an internal standard to the sample, allows only semi-quantitative determination of TIC concentration. This means that a quantitation limit for a particular compound cannot be determined. While the analysis method used here could have detected other chemical warfare agents (CWAs), including GB, GD, GF, HD and VX, the absence of these compounds from the samples cannot be related to a quantitation limit for each CWA. Additionally, regardless of quantitation limits, the presence of other chemicals in the sample could prevent the detection of a specific CWA if it were present at a low concentration and happened to co-elute from the GC column at a similar retention time as another compound present at a higher concentration than the CWA.

Table 7 GA Tentatively Identified Compounds

TIC	CAS #	RT (min)	D-25253-L (mg/mL)	D-25253-L-D (mg/mL)	D-35248-L (mg/mL)	D-29813-L (mg/mL)	D-51365-L (mg/mL)
Phosphoramidofluoric acid dimethyl- ethyl ester	358-29-2	6 00	4				
Unknown		6 61				8	9
Dimethyl dimethylphosphoramidate	597-07-9	6 70				10	10
Unknown phosphonate		7 01	5				
Unknown		7 11				9	9
Ethyl methyl N N-dimethylphosphoroamidate	135505-94-1	7 15	20			80	70
Phosphoramidocyanidic acid N N-dimethyl- methyl ester	63815-56-5	7 30	3				
N N-Dimethyl O O -diethyl phosphoramidate	2404-03-7	7 55	100	100	100	200	200
Triethyl phosphate	78-40-0	7 56			10		
GA	77-81-6	7 82	1 000	1 000	900	700	800
Bis(N N-dimethyl) O-ethyl phosphorodiamidate	2404-65-1	8 07	5		60	80	90
Thiazolo[3 2-a]pyridinium 2-carboxy-2 3-dihydro-8-hydroxy-5-methyl- hydroxide	23003-38-5	8 34	3				
Tetramethyl phosphorocyanidic diamide	14445-60-4	8 80	80	80	70	100	110
Unknown phosphate		10 85				10	
Unknown phosphate		10 96				9	10
Unknown phosphate		11 07				30	100
Unknown phosphate		11 08	60	80	200	200	90
Unknown phosphate		11 17	60	60	200	100	100
Unknown		11 49	10	10	20	80	80
Unknown		12 16	3				
Unknown		12 29	5			10	9
Unknown		12 39	3				
Unknown		12 85	7				
Heptadecanoic acid 16-methyl- methyl ester	5129-61-3	13 11	3				
Unknown		13 93	3				
Unknown		14 43				9	10
Unknown		14 66					10
Unknown		14 86	6				
Unknown		14 89	3				
GC/MS Data File			09030913 D	09030914 D	09030915 D	09030918 D	09030921 D

2 3 5 Agent purity and TICs for Lewisite liquid samples

Purity was determined following SOP HMRC IV-055. Samples were prepared by diluting 10 µL of neat agent in 10 mL of hexane. A 0.5 mL sample aliquot was then derivatized with 5.0 mL of 1% ethane thiol in 2,2,4-trimethylpentane (TMP) following procedures described in Edgewood Chemical and Biological Center (ECBC)-TR-531. The ethane thiol reacts with each chlorine functional group on the L1 and L2 forming an arsenic-sulfur bond, L3 is not derivatized as it does not contain a chlorine bonded to arsenic. Derivatization of L1 and L2 allows these compounds, which are thermally unstable, to be more readily analyzed by gas chromatography.

The derivatized samples were first analyzed by GC/MS to determine the retention time of L1, L2 and L3. The derivatized samples were then analyzed by GC/FID using a 10:1 split injection. A derivatized preparation blank was analyzed and used to establish a baseline response. Each purity sample was corrected using this blank baseline response. Purity was calculated as the Lewisite peak area (L1 or L2) divided by the total peak area response for the sample corrected for baseline response.

$$\% \text{ Purity} = \frac{\text{Lewisite Peak Area}}{(\text{Total Sample Peak Area Response} - \text{Preparation Blank Area Response})} \times 100$$

Test results are shown in Table 8. L1 purity ranged from 73.9% to 80.4%, L2 purity ranged from 13.0% to 16.6%, L3 was not identified during GC/MS analysis so a purity could not be determined. Interestingly, the summed purity for L1 and L2 was quite similar for all samples, with an average of 91.7% and a relative standard deviation of 1.2%. This indicates a strong inverse relationship between the amount of L1 and L2. RPD values for the field duplicate and batch duplicate were low for both L1 and L2, indicating good sample collection and analytical method reproducibility.

Table 8 Lewisite Purity

Sample ID	L1	L2	L3	L1 + L2	L1 RPD	L2 RPD
D-49221-L-01	77.6%	15.1%	ND	92.7%	1.1%	2.0%
D-49221-L-D	76.7%	14.8%	ND	91.5%		
D-79685-L-01	73.9%	16.6%	ND	90.6%	1.2%	5.5%
D-79685-L-01 DUP	74.8%	15.7%	ND	90.5%		
D-79693-L-01	80.1%	13.0%	ND	93.1%		
D-79697-L-01	80.4%	13.6%	ND	93.9%		
D-79699-L-01	74.3%	16.4%	ND	90.7%		
D-79700-L-01	76.7%	15.4%	ND	92.1%		
D-79701-L-01	77.3%	14.8%	ND	92.0%		
D-79703-L-01	76.5%	14.8%	ND	91.3%		
D-79705-L-01	77.4%	14.6%	ND	92.1%		
D-79711-L-01	75.1%	15.3%	ND	90.4%		
Average				91.7%		
RSD				1.2%		

ND = not detected RPD = relative percent difference RSD = relative standard deviation

Underivatized samples were also analyzed by GC/MS following guidelines of SOP HMRC-055. Prior to sample analysis, a GC/MS tune check was performed using DFTPP. Only a few peaks were detected for each sample. The mass spectra for each peak was compared to spectra from a NIST 2002 mass spectral library for tentative identification. Spectra with a quality match (Q) ≥ 75 were reported as the library identified compound. Because an internal standard was not added to these samples, a semi-quantitative TIC concentration was not calculated.

Test results are shown in Table 9. L1 and L2 were detected in all samples. The relative abundance of L1 to L2 is shown to be lower than what was observed for GC/FID analysis of the derivatized samples. Two factors may be affecting the abundance of L1. First, because underivatized L1 is thermally unstable, a greater proportion of L1 than L2 may have been lost in the heated GC injection port. Second, these samples were analyzed by split-less injection to aid in the detection of low concentration components. This resulted in a very large amount of L1 and L2 loaded onto the GC column which could have resulted in greater response saturation for L1 than L2, leading to a lower relative response. L1 and L2 were also detected as small "ghost" peaks in each of these samples, likely as a result of instrument overloading, and were not reported here.

L3 and arsenic trichloride were detected in most samples but at much lower levels than L1 and L2. While L3 was detected by this analysis, it was not detected in the derivatized samples, primarily due to the 10-fold dilution of the sample caused by derivatization procedure (0.5 mL of sample reacted with 5 mL of 1% ethane thiol in TMP).

While the analysis method used here could have detected other CWAs, including GA, GB, GD, GF, HD and VX, the absence of these compounds from the samples cannot be related to a quantitation limit for each CWA. Additionally, regardless of quantitation limits, the presence of other chemicals in the sample could prevent the detection of a specific CWA if it were present at a low concentration and happened to co-elute from the GC column at a similar retention time as another compound present at a higher concentration than the CWA.

Table 9 Lewisite Tentatively Identified Compounds

Compound	CAS #	RT (min)	D-49221-L-01 (Peak Area)	D-49221-L-D (Peak Area)	D-79685-L-01 (Peak Area)	D-79685-L-01 Dup (Peak Area)	D-79693-L-01 (Peak Area)	D-79697-L-01 (Peak Area)
Arsenic trichloride	007784-34-1	5 33	ND	716,742	1,238,970	1,229,140	849,492	831,435
Lewisite I	000541-25-3	7 41	49 227 400	57 454,900	58,768,100	57,306,300	58,021,400	56 035,900
Lewisite 2	040334-69-8	8 91	38 526 100	40,223 100	39 831,100	39 643,400	36,019 700	35 214,700
Lewisite 3	040334-70-1	9 95	619,148	673,802	1,003,250	932,951	ND	ND
GC/MS Data File			08190941 D	08190942 D	08190943 D	08190944 D	08190945 D	08190946 D
Compound	CAS #	RT (min)	D-79699-L-01 (Peak Area)	D-79700-L-01 (Peak Area)	D-79701-L-01 (Peak Area)	D-79703-L-01 (Peak Area)	D-79705-L-01 (Peak Area)	D-79711-L-01 (Peak Area)
Arsenic trichloride	007784-34-1	5 33	1 614 970	1,238 860	1 253,710	1,396 660	1 476,200	1 808 630
Lewisite I	000541-25-3	7 41	59 360,700	56,256 200	54,649,200	56,483 100	57,291,900	56,801,800
Lewisite 2	040334-69-8	8 91	38 746,500	35,865 100	35,006 000	36,471,000	36,425,500	36,858,800
Lewisite 3	040334-70-1	9 95	787,901	ND	556,240	570,466	587,049	660 127
GC/MS Data File			08190947 D	08190948 D	08190949 D	08190950 D	08190951 D	08190952 D

2 3 6 Headspace TIC VOCs for “transparency” ton sorbent tube samples

One solid sorbent tube sample was collected from each of the ten “transparency” ton container headspace for qualitative VOC identification and semi-quantitative analysis. Each sample sorbent tube was spiked with a solution of four internal standards, pentafluorobenzene, 1,4-difluorobenzene, chlorobenzene- d_5 and 1,4-dichlorobenzene- d_4 , from AccuStandard (catalog # M-8240/60-IS) with 200 ng of each IS added to each tube. Sorbent tubes were thermally desorbed and analyzed by GC/MS following guidelines of SOP HMRC-067. A Test Details sheet was also prepared. The suitability of this procedure for the analysis of VOCs was demonstrated by the analysis of a reference standard containing 53 VOCs (AccuStandard catalog # M-502A-R2) concurrent with sample analysis. Prior to the reference standard and sample analysis, a bromofluorobenzene standard was analyzed to verify proper MS operation. This standard met acceptance criteria.

Components in both samples and the VOC reference standard were qualitatively identified by mass spectral library match using Agilent ChemStation software designed for the analysis of TICs and an NIST 2002 mass spectral library. Semi-quantitative results of the VOC TICs were reported using ChemStation by comparison to internal standards. The sorbent tube that was used to collect the sample from ton container D39003 was somehow defective as it appears that no sample was collected on the tube. No internal standard was detected when the tube was desorbed. The tube was spiked a second time with IS and again, no IS was detected when the tube was desorbed. The client was notified of this situation immediately. Three equipment blanks were also collected and analyzed. See Table 10 for the identification of sorbent tubes used to sample each ton container and the GC/MS data file for the sorbent tube analysis.

Table 10 Ton Container ID and Sample Sorbent Tube ID

Transparency TC ID	Sorbent Tube ID	GC/MS Data File
D13754	MI-140318	07280906 D
D53763	MI-140313	07280909 D
D39003	MI-140594	07280911 D
D81037	MI-140317	07280913 D
D2425	MI-140320	07280915 D
D46304	MI-140316	07280917 D
D43593	MI-140599	07280919 D
D34998	MI-140312	07280921 D
D45358	MI-140595	07280923 D
D36234	MI-140315	07280925 D
Equipment Blank 1	MI-140593	07280927 D
Equipment Blank 2	MI-140314	07280929 D
Equipment Blank 3	MI-140311	07280931 D

Figure 2 shows the chromatogram of the VOCs collected from ton container D81037. This chromatogram is representative of what was collected from the other transparency ton containers.

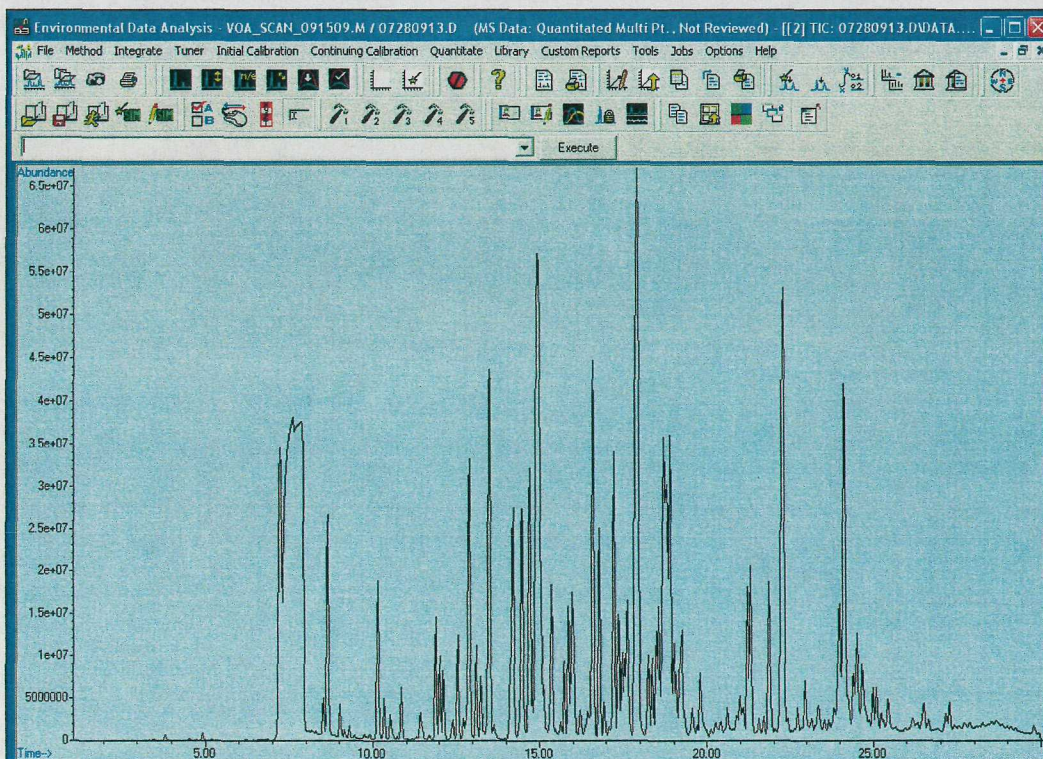


Figure 2. GC/MS Chromatogram of VOCs from TC D81037

Table 11 shows the results of the reference standard analysis. A 1.0- μ L spike of the solution resulted in 200 ng of each component loaded onto the sorbent tube. Not all components were detected, in particular some of the more volatile components, such as bromoform and methylene chloride. This was likely due to early elution from the GC column. The mass spectral library was unable to differentiate between the cis and trans isomers of 1,3-dichloro-1-propene. VOC mass was calculated based on a single point comparison against 200 ng of an internal standard and was reported to two significant figures. As can be seen from Table 11, the semi-quantitative results of the analysis resulted in detected values ranging from 22 ng to 450 ng with an average mass of 140 ng. These results indicate that the analysis method provided good component identification with semi-quantitative results ranging from a low of approximately 1/10 of the nominal mass to approximately 2x the nominal mass and average mass for all VOCs of 140 ng.

The data files from the sample analysis for VOCs were reviewed and the peaks with a response of at least 10% of the internal standard were selected.

Table 12 through Table 23 show the tentatively identified compounds and semi-quantitative results of the transparency ton container samples as well as three equipment blanks. VOC mass was calculated based on a single point comparison against 200 ng of an internal standard. VOC concentration ($\mu\text{g/L}$) was calculated to one or two significant figures based on 1 L of air being sampled through each sorbent tube. Note that the VOC mass (ng) values have not been expressed in significant figures. The library quality match value is reported for each compound. VOCs with Q values <75 should be treated as suspect. As there were a significant number of compounds in this category, it was deemed more valuable to provide the VOC names rather than try to categorize the compounds into specific chemical classes. In some instances, the same compound is identified at two different retention times. In these cases, typically one compound has a better library match (higher Q value) than the other and should be considered the more likely match. Significant overloading of the GC column occurred for several of the identified compounds, particularly acetone. As there was only a single sample collected, re-analysis with a sample split, e.g., a 1:10 split ratio, was not possible. In some instances, the peaks exhibiting poor chromatography needed to be manually integrated and the concentrations determined by comparison to the IS pentafluorobenzene. The sample from TC D13754 did not show response for any of the internal standards. An average pentafluorobenzene response was calculated from the other samples and used to calculate estimated TIC concentrations. Hexane is believed to be an artifact of sample preparation and is not actually present in the samples or equipment blanks.

The analysis of samples for TICs, when performed with the addition of an internal standard to the sample, allows only semi-quantitative determination of TIC concentration. This means that a quantitation limit for a particular compound cannot be determined. While the analysis method used here could have detected other CWAs, including GA, GB, GD, GF and HD, the absence of these compounds from the samples cannot be related to a quantitation limit for each CWA. Additionally, regardless of quantitation limits, the presence of other chemicals in the sample could prevent the detection of a specific CWA if it were present at a low concentration and happened to co-elute from the GC column at a similar retention time as another compound present at a higher concentration than the CWA.

Table 11 VOC Reference Standard Results

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value
1-Propene, 2-chloro-	6,189,010	6.14	22	000557-98-2	62
Ethene, 1,1-dichloro-	6,552,250	7.04	23	000075-35-4	97
Ethene, 1,2-dichloro-, (E)-	23,902,700	8.36	84	000156-60-5	97
Ethane, 1,1-dichloro-	18,083,700	9.14	63	000075-34-3	95
1,2-Dichloroethylene	43,648,100	10.10	150	000540-59-0	95
Trichloromethane	127,899,000	10.58	450	000067-66-3	96
1-Propene, 1,1-dichloro-	50,941,400	11.14	180	000563-58-6	95
Benzene	48,141,300	11.51	250	000071-43-2	96
Ethane, 1,2-dichloro-	20,008,100	11.64	100	000107-06-2	81
Trichloroethylene	31,478,300	12.56	160	000079-01-6	98
Propane, 1,2-dichloro-	31,029,300	13.05	160	000078-87-5	97
Methane, dibromo-	16,862,500	13.27	86	000074-95-3	96
Methane, bromodichloro-	22,892,100	13.47	120	000075-27-4	96

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value
1-Propene, 1,3-dichloro-, (E)-	30,713,500	14 26	160	010061-02-6	97
Toluene	60,526,200	14 81	310	000108-88-3	95
1-Propene, 1,3-dichloro-, (E)-	27,718,200	15 27	56	010061-02-6	97
Ethane, 1,1,2-trichloro-	30,581,000	15 63	62	000079-00-5	98
Tetrachloroethylene	36,072,600	15 78	73	000127-18-4	98
Propane, 1,3-dichloro-	32,097,900	15 98	65	000142-28-9	95
Methane, dibromochloro-	20,466,900	16 36	41	000124-48-1	98
Ethane, 1,2-dibromo-	15,049,400	16 66	30	000106-93-4	95
Ethylbenzene	97,535,400	17 62	200	000100-41-4	81
Benzene, 1,3-dimethyl-	121,415,000	17 84	250	000108-38-3	97
Styrene	123,036,000	18 68	250	000100-42-5	91
Methane, tribromo-	20,141,100	19 18	41	000075-25-2	98
Benzene, (1-methylethyl)-	68,877,700	19 33	140	000098-82-8	94
Ethane, 1,1,2,2-tetrachloro-	36,904,300	20 05	67	000079-34-5	97
Benzene, propyl-	153,439,000	20 17	280	000103-65-1	90
Benzene, 1,3,5-trimethyl-	132,257,000	20 51	240	000108-67-8	94
Benzene, 1-chloro-4-methyl-	62,088,100	20 69	110	000106-43-4	97
Benzene, tert-butyl-	75,191,400	21 19	140	000098-06-6	95
Benzene, 1,2,3-trimethyl-	75,488,500	21 31	140	000526-73-8	95
Benzene, 1-methyl-4-propyl-	81,072,200	21 65	150	001074-55-1	94
Benzene, 1-methyl-4-(1-methylethyl)-	75,735,700	21 92	140	000099-87-6	97
Benzene, 1,2-dichloro-	56,269,100	22 03	100	000095-50-1	98
Benzene, butyl-	85,651,300	22 78	160	000104-51-8	95
Benzene, 1,3-dichloro-	60,221,300	23 04	110	000541-73-1	98
Propane, 1,2-dibromo-3-chloro-	65,251,400	24 71	120	000096-12-8	99
Benzene, 1,2,4-trichloro-	65,414,300	26 46	120	000120-82-1	98
1,3-Butadiene, 1,1,2,3,4,4-hexachloro-	62,878,600	26 67	110	000087-68-3	99
Naphthalene	98,858,700	27 11	180	000091-20-3	95
Benzene, 1,2,3-trichloro-	114,928,000	27 69	210	000087-61-6	98
Average			140		

Table 12 Ton Container D13754 TIC VOCs

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc (µg/L)
Acetone	2,147,483,647	7.16	18,795	000067-64-1	80	19
2-Pentanol, 2-methyl-	51,181,200	14.98	448	000590-36-3	72	0.4
Propane, 2,2'-[methylenebis(oxy)]bis-	79,334,400	15.44	694	002568-89-0	83	0.7
2-Pentanone, 3-methyl-	59,787,900	15.65	523	000565-61-7	49	0.5
Silanol, trimethyl-, propanoate	4,664,740	15.79	41	016844-98-7	27	0.04
3-Pentanol, 2-methyl-	17,079,400	15.93	149	000565-67-3	83	0.1
1-Propanol, 2-(1-methylethoxy)-	28,795,200	16.35	252	003944-37-4	72	0.3
Propane, 2,2'-[ethylidenebis(oxy)]bis-	12,042,400	16.37	105	004285-59-0	90	0.1
2-Propanol, 1-(1-methylethoxy)-	4,747,550	16.50	42	003944-36-3	78	0.04
2-Propanol, 1-(1-methylethoxy)-	5,527,520	16.97	48	003944-36-3	53	0.05
3,7-Nonadien-2-one, 8-methyl-, (E)-	4,923,040	17.01	43	035408-14-1	43	0.04
Ethanone, 1-cyclopropyl-	4,168,180	17.02	36	000765-43-5	38	0.04
Furan, tetrahydro-2,2,4,4-tetramethyl-	6,496,260	17.88	57	003358-28-9	25	0.06
Ethosuximide	64,620,000	18.12	566	000077-67-8	47	0.6
4-Piperidinone, 1-methyl-	5,418,110	18.86	47	001445-73-4	50	0.05
Sulfurous acid, bis(1-methylethyl) ester	7,765,070	21.00	68	004773-13-1	64	0.07
Propane, 1-(1-methylethoxy)-	5,386,980	21.00	47	000627-08-7	42	0.05
Disulfide, bis(1-methylethyl)	5,746,870	22.12	50	004253-89-8	91	0.05

Table 13 Ton Container D53763 TIC VOCs

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc (µg/L)
Acetone	2,147,483,647	7 16	1,825	000067-64-1	64	2
Hexane	668,258,000	8 62	446	000110-54-3	91	0 4
Formic acid, 1-methylethyl ester	708,233,000	9 06	472	000625-55-8	91	0 5
2-Butanone	438,726,000	10 18	293	000078-93-3	50	0 3
Acetic acid, 1-methylethyl ester	1,707,270,000	11 45	1,139	000108-21-4	72	1
3-Methoxy-2,2-dimethyloxirane	626,927,000	11 50	418	026196-04-3	47	0 4
2-Pentanone, 3-methyl-	933,266,000	14 89	467	000565-61-7	83	0 5
1,3-Dioxane, 2,4-dimethyl-	1,373,140,000	15 36	688	000766-20-1	93	0 7
Propane, 2,2'-[ethylidenebis(oxy)]bis-	977,054,000	15 89	489	004285-59-0	83	0 5
Thiophene, 2-chloro-	723,849,000	17 18	363	000096-43-5	97	0 4
Ethosuximide	780,194,000	17 87	391	000077-67-8	47	0 4
Vinyl chloroacetate	545,410,000	18 40	273	002549-51-1	43	0 3
Propane, 2,2',2''-[methylidynetris(oxy)]tris-	821,802,000	21 86	349	004447-60-3	40	0 3

Table 14 Ton Container D81037 TIC VOCs

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc (µg/L)
Acetone	1,600,657,693	7 60	16,552	000067-64-1	80	17
1-Pentene, 2-methyl-	12,754,400	8 52	132	000763-29-1	91	0 1
Hexane	90,465,600	8 64	935	000110-54-3	94	0 9
2-Pentene, 2-methyl-	12,163,100	9 02	126	000625-27-4	90	0 1
5,9-Dodecadien-2-one, 6,10-dimethyl-, (E,E)-	72,052,200	10 14	745	1000132-10-9	78	0 7
2-Butanol	14,544,600	10 35	150	000078-92-2	83	0 2
Methane, bromochloro-	6,792,690	10 53	70	000074-97-5	97	0 07
Amylene Hydrate	6,933,370	11 43	72	000075-85-4	56	0 07
Thiophene	45,946,700	11 89	406	000110-02-1	97	0 4
2-Pentanone	29,883,000	12 02	264	000107-87-9	72	0 3
Furan, tetrahydro-2,5-dimethyl-	10,550,700	12 41	93	001003-38-9	90	0 09
Diethyl sulfide	38,776,700	12 56	343	000352-93-2	97	0 3
2-Pentanone	131,149,000	12 90	1,160	000107-87-9	72	1
3-Pentanone	35,763,000	13 12	316	000096-22-0	90	0 3
2-Butanol	22,259,000	13 26	197	000078-92-2	72	0 2
2-Butanone, 3,3-dimethyl-	206,763,000	13 51	1,828	000075-97-8	91	2
2-Pentanol, 2-methyl-	145,000,000	14 22	1,282	000590-36-3	90	1
3-Pentanone, 2-methyl-	145,925,000	14 71	1,290	000565-69-5	91	1
2-Pentanone, 3-methyl-	477,192,000	14 94	7,148	000565-61-7	64	7
3-Pentanol, 2-methyl-	63,807,700	15 36	956	000565-67-3	64	1

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc. (µg/L)
3-Hexanone	24,724,600	15 74	370	000589-38-8	90	0 4
2-Pentanol, 3-methyl-	41,156,500	15 87	617	000565-60-6	83	0 6
3,3-Dimethyl-2-pentanone	90,018,700	16 79	1,348	020669-04-9	53	1
Glutaconic acid	124,333,000	17 22	1,862	001724-02-3	38	2
2-Pentanone, 3-[(acetyloxy)methyl]-3,4-dimethyl-, (+ -)-	5,490,940	17 54	82	078641-04-0	50	0 1
2-Propenoic acid, 2-methyl-, 1,2-ethanediylbis(oxy-2,1-ethanediyl) ester	493,650,000	17 90	7,395	000109-16-0	47	7
Thiophene, 3-ethyl-	34,198,500	18 25	512	001795-01-3	97	0 5
Heptane, 2-methyl-3-methylene-	23,883,100	18 36	358	062187-11-5	47	0 4
4-Piperidinone, 1-methyl-	59,976,000	18 70	898	001445-73-4	58	0 9
Piperazine, 1-methyl-4-(2-phenoxyethyl)-	72,076,900	18 89	1,080	328002-65-9	53	1
3-Hexanone, 4,4-dimethyl-	6,847,710	19 07	103	019550-14-2	90	0 1
Ethosuximide	70,225,000	19 24	1,052	000077-67-8	53	1
2-Pentanone, 3-ethyl-3-methyl-	30,440,700	19 79	456	019780-65-5	78	0 5
Hexane, 3-bromo-	38,980,200	21 21	22	003377-87-5	59	0 02
4-Heptanone, 3-ethyl-	55,238,400	21 30	31	001528-25-2	52	0 03
Pentane, 3-ethyl-3-methyl-	82,928,600	21 86	47	001067-08-9	52	0 05
4-Heptanol, 4-methyl-	19,535,900	22 95	11	000598-01-6	59	0 01
Dimethyl-cyano-phosphine	13,849,500	23 35	8	031641-57-3	53	0 01
1,4-Dithiane	161,109,000	24 13	91	000505-29-3	97	0 09
Benzene, 1,2,4,5-tetramethyl-	13,848,500	24 41	8	000095-93-2	89	0 01
Benzene, 1,2,3,5-tetramethyl-	28,122,600	24 52	16	000527-53-7	83	0 02
Dodecane	11,428,800	25 00	6	000112-40-3	95	0 01
Benzene, 4-ethyl-1,2-dimethyl-	13,493,600	25 45	8	000934-80-5	91	0 01

Table 15 Ton Container D2425 TIC VOCs

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc (µg/L)
Propene	12,428,100	3 82	98	000115-07-1	90	0 1
Acetone	37,196,000	7 19	294	000067-64-1	72	0 3
Isopropyl Alcohol	93,552,000	7 32	739	000067-63-0	86	0 7
1-Pentene, 2-methyl-	2,995,900	8 55	24	000763-29-1	91	0 02
Hexane	84,413,000	8 67	667	000110-54-3	94	0 7
1-Propanol, 2-(1-methylethoxy)-	114,324,000	8 98	904	003944-37-4	78	0 9
Diisopropyl ether	114,324,000	9 00	904	000108-20-3	45	0 9
(Methylthio)-acetonitrile	76,879,000	9 05	608	035120-10-6	50	0 6
Propane, 1-(1-methylethoxy)-	27,644,500	10 38	219	000627-08-7	74	0 2
Acetic acid	23,845,200	11 31	188	000064-19-7	91	0 2
Acetic acid, 1-methylethyl ester	153,406,000	11 42	1,213	000108-21-4	83	1
3-Methoxy-2,2-dimethyloxirane	56,716,200	11 47	448	026196-04-3	47	0 4
Acetic acid, 1-methylethyl ester	51,481,900	11 50	407	000108-21-4	64	0 4
n-Propyl acetate	15,975,300	13 12	104	000109-60-4	78	0 1
1,3-Dioxane, 4-methyl-	3,888,550	13 20	25	001120-97-4	72	0 03
2-Propanol, 1-chloro-	36,003,700	14 02	234	000127-00-4	78	0 2
Propanoic acid, 1-methylethyl ester	47,218,500	14 16	307	000637-78-5	78	0 3
Methyl Isobutyl Ketone	19,639,500	14 46	128	000108-10-1	72	0 1
3-Pentanol, 2-methyl-	4,099,640	15 34	22	000565-67-3	64	0 02
Ethanol, 2-(2-chloroethoxy)-	12,407,200	15 46	66	000628-89-7	64	0 07
2-Propanol, 1-(1-methylethoxy)-	16,934,000	16 02	90	003944-36-3	83	0 09
Ether, 2-chloro-1-methylethyl isopropyl	61,162,300	16 38	326	098277-76-0	83	0 3
Methyl ethyl disulfide	12,114,700	17 18	65	020333-39-5	94	0 06
Methyl isopropyl disulphide	25,801,400	18 78	138	040136-65-0	91	0 1
Disulfide, ethyl 1-methylethyl	3,469,290	21 00	20	053966-36-2	91	0 02

Table 16 Ton Container D46304 TIC VOCs

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc (µg/L)
Acetone	80,832,500	7 19	595	000067-64-1	72	0 6
Isopropyl Alcohol	191,585,000	7 32	1,409	000067-63-0	86	1
Hexane	72,209,300	8 65	531	000110-54-3	94	0 5
1-Propanol	3,899,640	9 17	29	000071-23-8	78	0 03
Cyclopentane, methyl-	3,317,980	9 76	24	000096-37-7	91	0 02
2-Butanone	6,331,930	10 10	47	000078-93-3	72	0 05
Methane, bromochloro-	6,914,700	10 52	51	000074-97-5	97	0 05
2-Pentanol, 2-methyl-	6,803,870	14 12	40	000590-36-3	83	0 04
2-Hexanol	6,081,150	15 00	28	000626-93-7	83	0 03
4-Piperidinone, 1-methyl-	7,019,920	17 87	33	001445-73-4	43	0 03
Decane	16,439,200	20 26	78	000124-18-5	97	0 08
Undecane	13,443,900	22 72	64	001120-21-4	95	0 06
Dodecane	12,355,300	25 00	59	000112-40-3	96	0 06
Tridecane	7,150,170	27 27	34	000629-50-5	94	0 03

Table 17 Ton Container D43593 TIC VOCs

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc (µg/L)
Acetone	1,274,722,071	7 78	11,410	000067-64-1	59	11
Propane, 2-(ethenyloxy)-	577,584,036	8 07	5,170	000926-65-8	91	5
Acetic acid, 1-methylethyl ester	782,645,790	11 56	7,005	000108-21-4	64	7
Ethane, 1,2-dichloro-	134,839,000	11 61	40	000107-06-2	86	0 04
Propane, 2,2'-[methylenebis(oxy)]bis-	412,042,736	14 73	3,688	002568-89-0	83	4
1,4-Oxathiane	2,147,483,647	18 80	19,221	015980-15-1	95	19
Thiourea, ethyl-	196,496,000	19 40	57	000625-53-6	38	0 06
Chloroethyl 2-hexyl ether	453,116,353	21 48	4,056	1000131-99-1	53	4
Thiophene, tetrahydro-	115,323,000	21 51	32	000110-01-0	32	0 03
Bis(2-chloroethyl) ether	718,236,531	21 91	6,429	000111-44-4	91	6
1-Oxa-4-thiaspiro[4 4]nonane	97,869,600	22 33	27	000176-38-5	59	0 03
Thietane	159,693,276	22 58	1,429	000287-27-4	46	1
3,9-Dioxa-6-thiaundecane, 2,10-dimethyl-	124,004,000	29 70	35	097916-00-2	91	0 03

Table 18 Ton Container D34998 TIC VOCs

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc (µg/L)
Acetone	1,714,860,269	7 22	5,106	000067-64-1	64	5
Propane, 2-(ethenyloxy)-	48,927,100	8 06	146	000926-65-8	91	0 1
Hexane	81,616,900	8 64	243	000110-54-3	94	0 2
2-Ethylacrolein	47,975,800	10 12	143	000922-63-4	64	0 1
2-Buten-1-ol, acetate	56,642,900	10 16	169	000628-08-0	50	0 2
Acetic acid, 1-methylethyl ester	85,045,700	11 45	253	000108-21-4	64	0 3
1,3-Dioxane, 2,4-dimethyl-	60,127,300	15 36	389	000766-20-1	94	0 4
Propane, 2,2'-[ethylidenebis(oxy)]bis-	65,597,000	15 92	424	004285-59-0	83	0 4
3-Penten-2-one, 4-methyl-	49,963,600	16 23	323	000141-79-7	91	0 3
2-Butenal, 3-methyl-	34,520,800	16 38	223	000107-86-8	94	0 2
1,3-Butadiene, 1,4-dichloro-	37,728,800	16 78	244	002984-42-1	97	0 2
2-Octanol, (S)-	89,437,300	17 83	578	006169-06-8	43	0 6
Triethylene glycol	89,437,300	17 86	578	000112-27-6	47	0 6
Hydroperoxide, 1-methylpentyl	28,300,300	17 86	183	024254-55-5	50	0 2
Oxirane, 2-methyl-3-propyl-, cis-	20,237,100	17 87	131	006124-90-9	50	0 1
2-Nonanol	33,306,300	17 87	215	000628-99-9	47	0 2
Tetrahydrofuran, 2,2-dimethyl-	23,642,900	18 36	153	001003-17-4	17	0 2
Butyl-tert-butyl-isopropoxyborane	34,559,500	18 37	223	097782-82-6	25	0 2
1,4-Oxathiane	92,951,600	18 89	601	015980-15-1	95	0 6
4-Isopropoxy-2-butanone	73,642,400	19 50	476	032541-58-5	86	0 5
Ethanethioamide, N,N-dimethyl-	24,180,900	19 72	156	000631-67-4	10	0 2
Ether, 2-chloro-1-methylethyl isopropyl	62,221,900	19 92	439	098277-76-0	59	0 4
Propane, 1-(1-methylethoxy)-	24,071,400	19 96	170	000627-08-7	45	0 2
2-Chloroethyl methyl ether	46,430,600	20 52	328	000627-42-9	59	0 3
Propane, 2,2',2''-[methylidynetris(oxy)]tris-	56,026,000	21 87	396	004447-60-3	42	0 4
1,4-Dithiane	71,792,100	24 12	507	000505-29-3	97	0 5

Table 19 Ton Container D45358 TIC VOCs

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc (µg/L)
Propene	74,831,500	3.81	221	000115-07-1	90	0.2
Acetaldehyde	141,929,000	4.92	419	000075-07-0	56	0.4
Propane, 2-chloro-	67,001,100	6.81	198	000075-29-6	74	0.2
Acetone	2,027,772,105	8.06	6,500	000067-64-1	64	6
1-Pentene, 2-methyl-	144,238,000	8.49	426	000763-29-1	91	0.4
Hexane	935,795,000	8.61	2,764	000110-54-3	94	3
2,3-Dihydrofuran	69,237,000	8.83	204	001191-99-7	91	0.2
Formic acid, 1-methylethyl ester	349,810,000	9.05	1,033	000625-55-8	90	1
1-Propanol	174,150,000	9.43	514	000071-23-8	49	0.5
Acetaldehyde, chloro-	127,717,000	9.73	377	000107-20-0	97	0.4
Methyl vinyl ketone	138,274,000	9.94	408	000078-94-4	78	0.4
2-Butenal, 3-methyl-	1,074,130,000	10.12	3,172	000107-86-8	74	3
2-Butanol	155,198,000	10.44	458	000078-92-2	83	0.5
Trichloromethane	71,309,600	10.62	211	000067-66-3	91	0.2
Acetic acid, 1-methylethyl ester	5,003,340,000	11.52	619	000108-21-4	50	0.6
2-Propanol, 1-chloro-	3,173,510,000	14.15	393	000127-00-4	72	0.4
Propane, 2,2'-[methylenebis(oxy)]bis-	3,300,220,000	14.78	409	002568-89-0	83	0.4
2-Pentanone, 3-methyl-	1,726,570,000	14.94	727	000565-61-7	94	0.7
2-Hexanol	710,039,000	15.06	299	000626-93-7	78	0.3
1,3-Dioxane, 2,4-dimethyl-	2,107,410,000	15.37	888	000766-20-1	80	0.9
Propane, 2,2'-[ethylidenebis(oxy)]bis-	918,747,000	15.95	387	004285-59-0	83	0.4
2-Propanol, 1-(1-methylethoxy)-	828,816,000	16.07	349	003944-36-3	83	0.3
1-Propanol, 2-(1-methylethoxy)-	629,983,000	16.60	265	003944-37-4	53	0.3
2-Ethyl-trans-2-butenal	443,733,000	17.03	187	063883-69-2	94	0.2
Propanol, methoxy-, acetate	442,211,000	17.31	186	084540-57-8	42	0.2
2-(2-Hexyloxyethoxy)ethanol	3,170,580,000	17.88	1,336	000112-59-4	47	1
2-Propoxy-tetrahydropyran	469,496,000	18.31	198	006581-64-2	59	0.2
Propane, 1-chloro-2-nitro-	698,155,000	18.39	294	002425-66-3	56	0.3
Furan, tetrahydro-2,2,4,4-tetramethyl-	707,752,000	18.70	298	003358-28-9	59	0.3
1,4-Oxathiane	985,676,000	18.89	415	015980-15-1	96	0.4
4-Isopropoxy-2-butanone	437,992,000	19.52	185	032541-58-5	50	0.2
Diisopropyl ether	933,357,000	19.93	255	000108-20-3	53	0.3
Propane, 2,2',2''-[methylidynetris(oxy)]tris-	1,204,720,000	21.88	329	004447-60-3	50	0.3
Benzene, 1-ethyl-2,3-dimethyl-	673,708,000	22.71	184	000933-98-2	81	0.2
1,4-Dithiane	673,111,000	24.13	184	000505-29-3	98	0.2

Table 20 Ton Container D36234 TIC VOCs

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc (µg/L)
Acetaldehyde	17,640,800	4.92	176	000075-07-0	64	0.2
Acetone	1,891,766,084	8.05	18,839	000067-64-1	72	19
Hexane	75,726,900	8.62	754	000110-54-3	94	0.8
Formic acid, 1-methylethyl ester	83,447,800	9.04	831	000625-55-8	72	0.8
Diisopropyl ether	28,360,900	9.06	282	000108-20-3	64	0.3
1-Propanol, 2-(1-methylethoxy)-	65,324,900	9.10	651	003944-37-4	72	0.7
2-Butanone	56,013,100	10.15	558	000078-93-3	59	0.6
Acetic acid, 1-methylethyl ester	191,850,000	11.44	1,911	000108-21-4	83	2
Thiophene	18,222,100	11.88	108	000110-02-1	97	0.1
2-Pentanone	50,280,700	12.92	298	000107-87-9	91	0.3
2-Butanone, 3,3-dimethyl-	41,079,300	13.51	243	000075-97-8	64	0.2
Propanoic acid, 1-methylethyl ester	25,267,400	14.19	150	000637-78-5	83	0.1
3-Pentanone, 2-methyl-	18,404,200	14.70	109	000565-69-5	49	0.1
Propane, 2,2'-[methylenebis(oxy)]bis-	49,011,100	14.75	290	002568-89-0	59	0.3
2-Pentanone, 3-methyl-	97,410,000	14.88	756	000565-61-7	90	0.8
1,3-Dioxane, 2,4-dimethyl-	121,109,000	15.35	940	000766-20-1	91	0.9
Propane, 2,2'-[ethylidenebis(oxy)]bis-	68,838,400	15.93	534	004285-59-0	90	0.5
3-Heptanol	13,490,900	16.59	105	000589-82-2	38	0.1
Ethosuximide	60,818,600	17.87	472	000077-67-8	50	0.5
3,7-Dimethyloctyl ethylphosphonofluoridoate	30,884,200	17.88	240	1000298-33-6	40	0.2
Monochloroacetic acid isopropyl ester	23,282,300	18.39	181	000105-48-6	74	0.2
1,4-Oxathiane	19,449,000	18.87	151	015980-15-1	96	0.2
Propane, 2,2',2''-[methylidynetris(oxy)]tris-	69,275,400	21.87	325	004447-60-3	50	0.3

Table 21 VOC Equipment Blank 1

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc (µg/L)
Acetone	10,703,200	7.19	87	000067-64-1	72	0.09
Isopropyl Alcohol	6,861,120	7.33	56	000067-63-0	72	0.06
Hexane	96,795,100	8.67	789	000110-54-3	94	0.8
Acetic acid, 1-methylethyl ester	3,611,470	11.47	29	000108-21-4	83	0.03
1,4-Dithiane	14,387,600	24.12	70	000505-29-3	97	0.07

Table 22 VOC Equipment Blank 2

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc (µg/L)
Acetone	82,224,300	7.19	108	000067-64-1	72	0.1
Isopropyl Alcohol	41,017,300	7.33	54	000067-63-0	78	0.05
Hexane	662,862,000	8.67	868	000110-54-3	94	0.9
1,4-Oxathiane	149,409,000	18.87	78	015980-15-1	96	0.08
Phenol	81,258,400	22.95	38	000108-95-2	60	0.04
Diethyl Phthalate	80,673,900	23.11	38	000084-66-2	98	0.04
1,4-Dithiane	133,925,000	24.16	63	000505-29-3	98	0.06

Table 23 VOC Equipment Blank 3

Tentatively Identified Compound	Area Counts	RT (min)	Mass (ng)	CAS #	Q Value	Conc (µg/L)
Acetone	57,594,000	7.19	80	000067-64-1	80	0.08
Isopropyl Alcohol	21,436,600	7.33	30	000067-63-0	78	0.03
Pentane, 2-methyl-	20,227,700	7.80	28	000107-83-5	91	0.03
Methylene Chloride	20,108,900	8.01	28	000075-09-2	87	0.03
Hexane	1,089,950,000	8.67	1,510	000110-54-3	94	1.5
Cyclopentane, methyl-	15,592,700	9.76	22	000096-37-7	91	0.02
1,4-Oxathiane	70,451,600	18.87	35	015980-15-1	96	0.04
Phenol	58,034,000	22.95	26	000108-95-2	76	0.03
Diethyl Phthalate	59,563,100	23.12	27	000084-66-2	98	0.03

2.3.7 HRA Metals for GA liquid, Lewisite liquid and Lewisite sludge samples

Samples were prepared (digested) at the HMRC and analyzed at Battelle's laboratory at King Ave., Columbus, OH. Sample preparation and analysis followed SOP HMRC X-241-00, which was based on TE-LOP-557 and TE-LOP-584. See the Test Plan (Appendix C) for method QA/QC criteria. Samples were prepared in two batches, one containing the liquid Lewisite and GA samples and the other containing all of the Lewisite sludge samples. For all sample types, approximately 0.1 g of sample was weighed and digested using 10 mL of a nitric acid / hydrochloric acid solution (4.5 HNO₃, 4.5 DI water, 1 HCl). Following microwave digestion, samples were diluted to 100 mL in DI water. Samples were analyzed by inductively coupled mass spectrometry (ICP-MS) for the 20 HRA metals. Calibrations for the metals varied but typically comprised 9 levels ranging from 0.1 to 50 µg/L. Due to varying metal concentrations, samples were analyzed at multiple dilutions, ranging from undiluted up to a factor of 100,000 in the case of mercury analysis of sludge samples. Iron was not originally identified as a metal of interest (not an HRA metal), however, in an attempt to better characterize the composition of only the Lewisite sludge, iron was also analyzed. No LCS or MS/MSD data are available for iron since it was not included as a target compound.

Results for GA liquid are shown in Table 24. The RPD is calculated for sample D25253-L-01 and the field duplicate collected from the same tank, D25253-L-D. While several of the metals

exceed 25% RPD, this is not unexpected for a ton container field duplicate, where collection of collocated sample is difficult and some material inhomogeneity may exist

Table 24 HRA Metals in GA Liquid

Metal	D25253-L-01 (mg/Kg)	D25253-L-D (mg/Kg)	RPD	D29813-L-01 (mg/Kg)	D35248-L-01 (mg/Kg)	D51365-L-01 (mg/Kg)
Aluminum	41.5	28.3	38%	40.3	30.3	37.3
Antimony	0.54	0.49	8.5%	0.23	334	316
Arsenic	30.6	34.8	13%	29.8	49.6	32.9
Barium	0.38	1.73	128%	0.62	0.39	0.36
Beryllium	<0.05	<0.05	NA	<0.05	<0.05	<0.05
Boron	137	80.7	52%	112	113	95.6
Cadmium	<0.05	<0.05	NA	<0.05	<0.05	<0.05
Chromium	2.28	1.97	15%	2.57	1.25	1.29
Cobalt	0.054	<0.05	NA	<0.05	<0.05	<0.05
Copper	36.9	6.32	142%	10.3	0.82	1.72
Lead	32.6	4.13	155%	18.7	0.27	0.77
Manganese	2.18	0.31	150%	1.32	0.13	0.21
Mercury	0.29	7.91	186%	0.49	10.4	1.81
Nickel	0.90	0.46	64%	0.70	0.09	0.19
Selenium	<0.05	<0.05	NA	<0.05	0.07	0.09
Silver	<0.05	<0.05	NA	<0.05	0.12	0.12
Thallium	<0.05	<0.05	NA	<0.05	<0.05	<0.05
Tin	0.38	5.69	175%	<0.25	0.27	6.75
Vanadium	9.43	9.73	3.1%	11.5	13.9	11.0
Zinc	52.1	73.3	34%	43.1	28.3	36.7

Results for Lewisite liquid are shown in Table 25. The RPD is calculated for sample D49221-L-01 and the field duplicate collected from the same tank, D49221-L-D. Most of the detected metals had an RPD below 25% indicating excellent sample collection reproducibility. Results for Lewisite sludges are shown in Table 26. A field duplicate was not collected for the sludges.

The quality control sample results for the liquid agent preparation batch (which included both GA and Lewisite liquid samples) are shown in Table 27. Note that a small amount of mercury (1.49 µg/L) was detected in the preparation blank. This concentration of mercury is not significant relative to the amount of mercury found in the Lewisite liquid samples. This amount of mercury is similar to, or higher than, the amount of mercury found in some of the GA liquid samples. Based on the overall data quality objectives of this testing, the mercury in the preparation blank does not impact the data. Also of note is the elevated recovery of vanadium for the laboratory control sample (LCS-L). The cause of this high recovery is not readily apparent but is not expected to impact the data, as higher concentrations of vanadium than what was used for the LCS spike were found in all samples. Several metals had RPDs above 20% for the sample duplicate, however, for these metals the concentration in the samples typically was not >100 times the instrument detection limit (IDL). Matrix spike (MS) and matrix spike duplicate (MSD) recoveries were typically quite good, as were RPD values between the MS and MSD. Recoveries were not calculated for samples where the spike concentration was <10% of the native metal concentration.

The quality control sample results for the Lewisite sludge preparation batch are shown in Table 28. Only iron was detected in the blank sample but at concentrations well below those found in the sample, there is no impact to the data. The recovery of several metals in the LCS were above 125%, in particular aluminum, boron, copper and vanadium. Cross contamination from another sample seems unlikely as the recovery of arsenic and mercury, which are very high in the samples, was acceptable in the LCS. As there was no evidence of contamination in the preparation blank, these elevated recoveries may have been due to a random introduction of contamination. Comparing LCS metals concentrations to sample concentrations, aluminum, boron and vanadium may be biased high in the samples but since the preparation blank did not show evidence of contamination this is only speculation. The elevated LCS recoveries do not represent a significant impact to the data. The sample and sample duplicate had RPD values >25% for several of the metals. As the samples were tar-like in nature, there may be some inherent inhomogeneity in the sample. Additionally, it was not feasible to homogenize or grind the sludge samples prior to collection of subsamples for metals analysis. Recoveries for many metals in the MS and MSD samples were below 75%. This was likely due to the sample inhomogeneity exhibited with the duplicate samples.

Table 25 HRA Metals in Lewisite Liquid

Metal	D49221-L-01 (mg/Kg)	D49221-L-D (mg/Kg)	RPD	D79685-L-01 (mg/Kg)	D79693-L-01 (mg/Kg)	D79697-L-01 (mg/Kg)	D79699-L-01 (mg/Kg)	D79700-L-01 (mg/Kg)	D79701-L-01 (mg/Kg)	D79703-L-01 (mg/Kg)	D79705-L-01 (mg/Kg)	D79711-L-01 (mg/Kg)
Aluminum	28.4	31.9	12%	29.8	44.0	37.0	31.8	31.5	37.0	32.8	34.1	34.5
Antimony	298	316	5.8%	292	320	326	338	325	323	296	312	292
Arsenic	330.000	340.000	3.0%	321.000	308.000	316.000	335.000	323.000	331.000	323.000	308.000	305.000
Barium	0.36	0.32	11%	0.36	0.50	0.37	0.37	0.16	0.59	0.37	0.34	0.36
Beryllium	<0.05	<0.05	NA	<0.05	0.10	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Boron	110	93.1	17%	88.1	78.8	86.6	104	113	110	103	103	93
Cadmium	<0.05	<0.05	NA	<0.05	0.10	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Chromium	1.73	1.25	32%	1.45	1.46	1.17	1.17	1.21	1.59	1.25	1.27	1.30
Cobalt	<0.05	<0.05	NA	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Copper	0.43	1.20	95%	0.43	0.27	2.35	2.27	0.33	1.76	0.26	0.29	0.59
Lead	0.27	0.29	6.6%	0.28	0.34	0.23	0.23	0.18	0.87	0.21	0.26	0.28
Manganese	0.23	0.20	16%	0.17	0.25	0.13	0.11	0.35	0.16	0.13	0.13	0.14
Mercury	158	174	10%	528	48.4	115	315	112	161	137	169	173
Nickel	0.37	0.15	82%	0.17	0.31	0.08	0.06	0.14	0.20	0.07	0.07	0.84
Selenium	<0.05	<0.05	NA	<0.05	6.36	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Silver	0.14	0.12	17%	0.11	0.35	0.12	0.12	0.11	0.12	0.11	0.10	0.10
Thallium	<0.05	<0.05	NA	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Tin	<0.25	0.30	NA	<0.25	0.40	<0.25	<0.25	<0.25	0.26	<0.25	0.80	0.95
Vanadium	11.0	12.8	16%	12.7	15.3	15.1	16.9	11.8	14.1	14.4	14.3	14.6
Zinc	34.6	36.1	4.3%	36.1	49.0	51.1	36.9	41.5	30.8	72.0	32.9	58.6

Table 26 HRA Metals in Lewisite Sludge

Metal	D49221-S-01 (mg/Kg)	D79685-S-01 (mg/Kg)	D79693-S-01 (mg/Kg)	D79697-S-01 (mg/Kg)	D79699-S-01 (mg/Kg)	D79700-S-01 (mg/Kg)	D79701-S-01 (mg/Kg)	D79703-S-01 (mg/Kg)	D79705-S-01 (mg/Kg)	D79711-S-01 (mg/Kg)
Aluminum	207	146	91 8	57 0	190	59 9	69 8	54 5	63 6	61 6
Antimony	1 960	2 030	1 870	2 440	2 960	3 250	3 450	2 960	2 690	3 240
Arsenic	161 000	200 000	211 000	205 000	149 000	99 600	135 000	156 000	191 000	146 000
Barium	0 42	0 89	0 63	1 77	0 31	0 50	0 33	0 86	0 36	0 50
Beryllium	<0 05	0 19	<0 05	0 10	0 09	0 36	0 06	0 07	<0 05	<0 05
Boron	186	127	160	143	183	177	189	138	168	194
Cadmium	0 19	0 43	0 10	0 12	0 26	0 59	0 73	0 15	0 21	0 24
Chromium	7 41	9 32	22 9	9 65	9 28	72 5	95 8	6 03	4 80	5 71
Cobalt	0 63	0 90	0 61	0 64	0 87	3 35	1 50	0 37	0 28	0 28
Copper	137	176	184	93 8	235	863	1120	157	197	312
Iron	9 250	8 740	22 300	11 600	6 210	39 100	5 180	3 070	4 330	3 020
Lead	127	98 8	95 6	745	616	2 600	320	523	25 3	41 7
Manganese	54 7	47 8	28 4	57 4	35 0	186	22 9	17 0	15 0	12 7
Mercury	173 000	181 000	131 000	291 000	428 000	493 000	452 000	428 000	338 000	437 993
Nickel	4 76	6 11	14 8	6 39	5 45	47 5	45 1	6 23	3 17	3 25
Selenium	3 56	3 29	7 52	7 09	3 63	7 79	10 2	5 90	7 56	9 12
Silver	0 10	0 35	0 17	0 12	0 08	0 12	0 15	0 08	0 09	0 09
Thallium	0 41	0 66	0 25	0 47	0 56	0 63	0 55	0 53	0 42	0 51
Tin	10 4	12 0	10 3	9 06	10 3	7 36	16 6	10 6	15 8	14 3
Vanadium	18 8	20 2	16 1	18 3	17 7	17 2	21 2	19 1	21 5	22 3
Zinc	293	276	291	223	323	815	1194	356	271	334

Table 27 Quality Control Samples for the GA & Lewisite Liquid Preparation Batch

Metal	Prep Blank-L (mg/Kg)	LCS-L (mg/Kg)	Spike Conc (mg/Kg)	LCS Rec	D79685-L-01 (mg/Kg)	D79685-L-01 DUP (mg/Kg)	DUP RPD	D79693-L-01 (mg/Kg)	D79693-L-01 MS (mg/Kg)	D79693-L-01 MSD (mg/Kg)	MS Rec	MSD Rec	MS/MSD RPD
Aluminum	<5.00	5.58	5	112%	29.8	36.0	19%	44.0	52.3	46.7	165%	53%	103%
Antimony	0.06	0.55	0.5	111%	292	285	2.3%	320	290	325	ND	ND	NA
Arsenic	<5.00	0.53	0.5	105%	321.000	304.000	5.4%	308.000	305.000	303.000	ND	ND	NA
Barium	<0.05	0.59	0.5	119%	0.36	0.71	66%	0.50	0.93	1.02	86%	104%	19%
Beryllium	<0.05	0.52	0.5	103%	<0.05	0.33	NA	0.10	0.60	0.60	100%	100%	0%
Boron	<5.00	6.47	5	129%	88.1	87.3	1.0%	78.8	108.7	124.2	ND	ND	NA
Cadmium	<0.05	0.54	0.5	108%	<0.05	0.38	NA	0.10	0.63	0.58	106%	96%	10%
Chromium	<0.05	0.53	0.5	107%	1.45	1.62	11%	1.46	2.04	1.95	115%	97%	17%
Cobalt	<0.05	0.54	0.5	109%	<0.05	0.38	NA	<0.05	0.56	0.53	111%	106%	5.1%
Copper	<0.05	0.49	0.5	97%	0.43	0.67	43%	0.27	0.80	0.73	106%	93%	14%
Lead	<0.05	0.52	0.5	103%	0.28	0.73	88%	0.34	1.01	0.82	134%	96%	33%
Manganese	<0.05	0.56	0.5	112%	0.17	0.57	108%	0.25	0.85	0.76	120%	102%	16%
Mercury	1.49	0.61	0.5	122%	528	518	1.9%	48.4	45.8	40.8	ND	ND	NA
Nickel	<0.05	0.51	0.5	101%	0.17	0.49	98%	0.31	0.87	0.71	113%	81%	33%
Selenium	<0.05	0.63	0.5	126%	<0.05	0.33	NA	6.36	7.66	8.91	ND	ND	NA
Silver	<0.05	0.52	0.5	104%	0.11	0.48	126%	0.35	0.83	0.86	96%	102%	5.5%
Thallium	<0.05	0.53	0.5	106%	<0.05	0.39	NA	<0.05	0.55	0.53	110%	106%	3.9%
Tin	<0.25	2.69	2.5	107%	0.15	3.66	184%	0.40	5.49	2.84	204%	98%	70%
Vanadium	<0.05	1.28	0.5	257%	12.7	13.5	6.2%	15.3	13.8	16.3	ND	ND	NA
Zinc	<0.5	4.88	0.5	98%	36.1	64.2	56%	49.0	43.2	48.5	ND	ND	NA

NA = not applicable

ND = not determined - spike concentration < 10% of the native metal concentration

Table 28 Quality Control Samples for the Lewisite Sludge Preparation Batch

Metal	Prep Blank-S (mg/Kg)	LCS-S (mg/Kg)	Spike Conc (mg/Kg)	LCS Rec	D49221-S-01 (mg/Kg)	D49221-S-01 DUP (mg/Kg)	DUP RPD	D79685-S-01 (mg/Kg)	D79685-S-01 MS (mg/Kg)	D79685-S-01 MSD (mg/Kg)	MS Rec	MSD Rec	MS/MSD RPD
Aluminum	<5 00	65 0	5	1300%	207	191	8 0%	146	229	77 4	ND	ND	NA
Antimony	<0 05	0 75	0 5	151%	1 960	2 370	19%	2 030	1 990	2 060	ND	ND	NA
Arsenic	<25 0	0 52	0 5	104%	161 000	153 000	5 1%	200 000	199 000	183 000	ND	ND	NA
Barium	<0 05	0 69	0 5	139%	0 42	0 77	60%	0 89	0 84	1 47	-10%	116%	238%
Beryllium	<0 05	0 44	0 5	89%	<0 05	0 28	NA	0 19	0 43	0 38	47%	37%	24%
Boron	<5 00	154	5	3075%	186	192	3 3%	127	176	124	ND	ND	NA
Cadmium	<0 05	0 48	0 5	96%	0 19	0 43	76%	0 43	0 65	0 69	44%	52%	19%
Chromium	<0 05	2 17	0 5	434%	7 41	9 00	19%	9 32	8 77	11 2	ND	ND	NA
Cobalt	<0 05	0 51	0 5	102%	0 63	0 97	42%	0 90	1 19	1 09	59%	39%	40%
Copper	<0 05	18 9	0 5	3777%	137	153	11%	176	159	193	ND	ND	NA
Iron	70	NP	NA	NA	9 250	7 390	22%	NP	NP	NP	NA	NA	NA
Lead	<0 05	0 69	0 5	137%	127	123	3 2%	98 8	91 1	33 2	ND	ND	NA
Manganese	<0 05	0 72	0 5	143%	54 7	44 1	22%	47 8	49 8	43 8	ND	ND	NA
Mercury	<1 00	0 56	0 5	112%	173 000	164 000	5 3%	181 000	192 000	173 000	ND	ND	NA
Nickel	<0 05	0 84	0 5	167%	4 76	6 47	30%	6 11	5 46	7 18	ND	ND	NA
Selenium	<0 05	0 47	0 5	94%	3 56	5 94	50%	3 29	3 69	3 70	79%	82%	3 9%
Silver	<0 05	0 52	0 5	103%	0 10	0 41	121%	0 35	0 69	0 63	69%	57%	20%
Thallium	<0 05	0 47	0 5	95%	0 41	0 81	66%	0 66	0 98	0 96	64%	60%	7 1%
Tin	<0 25	2 99	2 5	120%	10 4	10 2	1 5%	12 0	12 7	12 6	30%	25%	18%
Vanadium	<0 05	22 6	0 5	4521%	18 8	19 4	3 1%	20 2	19 5	17 9	ND	ND	NA
Zinc	<0 5	30 4	0 5	608%	293	313	6 6%	276	238	265	ND	ND	NA

NA = not applicable

ND = not determined - spike concentration < 10% of the native metal concentration

NP = not prepared

2 4 Lewisite Sludge Solubility Test

A test was performed to determine the solubility of sludge from Lewisite ton containers in three different solvents. Samples were prepared in a ratio of 1 g of sludge to 125 mL of solvent. The three solvents that were tested were water, 20% acetic acid and 7 M nitric acid. Sample D-79711-S-01 was used for the solubility testing. This sample contained a Hg content of 438,000 mg/Kg.

Approximately 0.1 g of sample was weighed into a 30-mL polypropylene container. 12.5 mL of test solvent was added and the solution mixed end-over-end for 2 hr using a rotary mixer. Samples were filtered through a glass fiber filter (GF/F), with 3 x 10 mL rinses of water used to transfer sample from the bottles to the filters. The filters dried in an oven at 103 -105°C consistent with Standard Methods 2540D. The amount of solids retained on the filter were determined gravimetrically.

The amount of undissolved solids retained by the filters are shown in Table 29, calculated as the final filter and dish weight minus the initial filter and dish weight. The percent undissolved solids was calculated as the ratio of the undissolved solids weight to the sample weight. The weight of dissolved solids was calculated as the difference of the sample weight and the undissolved solids weight.

Table 29 Weights of Undissolved and Dissolved Lewisite Sludge

Sample ID	Initial Filter & Dish Wt. (g)	Sample Weight (g)	Solvent Type	Final Filter & Dish Wt. (g)	Undissolved Solids (g)	Percent Undissolved Solids	Dissolved Solids (g)	Undissolved Solids RPD
S-W-1	37.9908	0.1014	DI Water	37.9988	0.0080	7.9%	0.0934	13%
S-W-2	37.7607	0.1069	DI Water	37.7677	0.0070	6.5%	0.0999	
S-A-1	38.8703	0.1014	20% Acetic Acid	38.8999	0.0296	29.2%	0.0718	58%
S-A-2	35.6003	0.0981	20% Acetic Acid	35.6542	0.0539	54.9%	0.0442	
S-N-1	36.5871	0.0970	7.0 M Nitric Acid	36.6060	0.0189	19.5%	0.0781	2.1%
S-N-2	36.3712	0.0991	7.0 M Nitric Acid	36.3897	0.0185	18.7%	0.0806	

Surprisingly, water appeared to dissolve the greatest amount of sludge, resulting in lower percent undissolved solids, i.e., more dissolved solids, than 20% acetic acid and 7.0 M nitric acid. This may have been due to the apparent ability of water to better disperse the sludge material compared to 7.0 M nitric acid and 20% acetic acid. This is shown in Figure 3 and Figure 4, where the sludge from sample SW-1 appears to be comprised of finer material than for sample SN-2. While the RPD for the water and nitric acid samples was low, the RPD for the acetic acid samples was high, indicating possible differences between the two sludge subsamples.

The three blank samples, one prepared with each solvent type, showed an average weight loss upon drying of 8.3 mg. As the filters were not conditioned prior to use, this may have been due to loss of water or other volatiles from the filters, this was a deviation from 2540D. This means

that the reported undissolved solids may be biased slightly low, however, the relative results would not change dramatically.

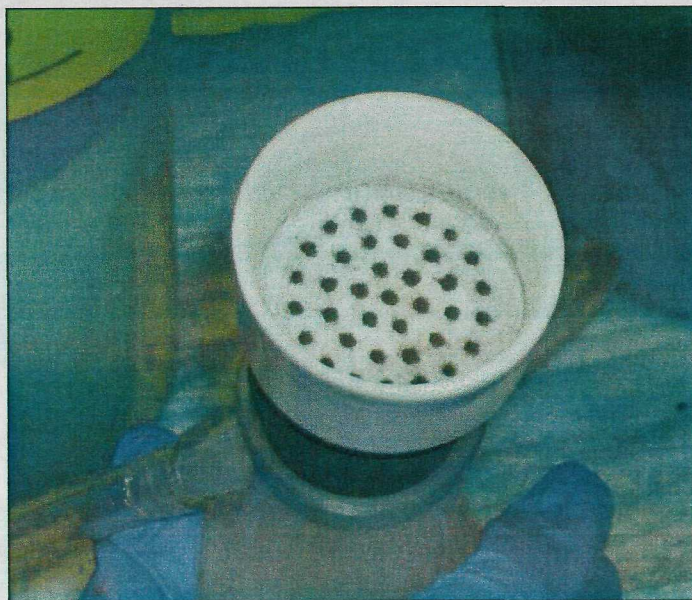


Figure 3. Filter SW-1

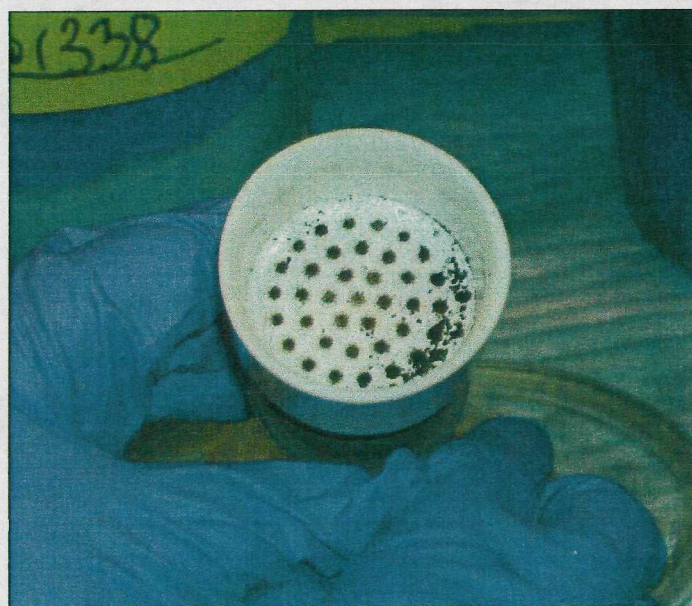


Figure 4. Filter SN-2

The filtrate from each sample was then analyzed for L1 as its ethanethiol derivative by GC/MS. The analytical method discussed in Section 2.5.2 below was used. Results of the analyses are

shown in Table 30. While there was a difference in the amount of solids dissolved by the water and 20% acetic acid, the amount of L1 in the rinsate was very similar. Significantly less L1 was detected in the 7.0 M nitric acid rinsate, likely due to the oxidation of L1 by the nitric acid. The RPD for L1 measured in the filtrate for each pair of subsamples was low, indicating uniform concentration of L1 in the sludge. The RPD between SN-2 and its duplicate was 5.3%, indicating lower variability in the sample preparation. The water and acetic acid samples were diluted 1000:1 prior to analysis and the nitric acid samples were diluted 100:1 prior to analysis. Because of these large dilutions, matrix spike recoveries could not be determined.

A qualitative analysis on the relative abundance of L2 was performed (L3 was not detected). The peak area of L2 was compared to the peak area of L1 in each sample. The results were expressed as a percentage of L2 relative to L1, shown in Table 30. Reviewing the purity data reported in Section 2.3.5, it appears that the relative abundance in neat agent of L2 (~15%) to L1 (~75%) is about 20%. A slightly lower ratio of L2 to L1 is seen for the water rinsate samples (7.7% and 11%) while the acetic acid rinsate had ratios close to 20%. The lower L2 percentage for the water rinsates may be related to poor water solubility of L2 while the 20% acetic acid may better solubilize L2. The nitric acid rinsate samples showed a very high amount of L2 relative to L1 (270% and 320%). These high ratios are due to a lower L1 response relative to L2. This indicates that, under these test conditions, 7.0 M nitric acid reacted more readily with L1 than L2. Taking sample dilutions into account, the L2 response was similar for the acetic acid and nitric acid rinsate samples.

Table 30 L1 and L2 in Sludge Filtrate

Data File Name	Sample ID	L1 Sample Conc (µg/L)	Filtrate Vol (L)	L1 Sample Mass (mg)	Spike Recovery	Sample Mass RPD	Area L2 / Area L1*
08270914.D	QC-W	<50	0.0425	<0.0021			
08270915.D	QC-A	<50	0.0425	<0.0021			
08270916.D	QC-N	<50	0.0425	<0.0021			
08270918.D	S-W-1	793,000	0.0425	34		15%	7.7%
08270925.D	S-W-2	684,000	0.0425	29			11%
08270921.D	S-A-1	733,000	0.0425	31		20%	23%
08270928.D	S-A-2	597,000	0.0425	25			21%
08270922.D	S-A-1 MS	719,000	0.0425	31	NA		22%
08270923.D	S-A-1 MSD	709,000	0.0425	30	NA		24%
08270924.D	S-N-1	70,300	0.0425	3.0		13%	270%
08270929.D	S-N-2	61,500	0.0425	2.6			320%
08270930.D	S-N-2 DUP	58,300	0.0425	2.5			320%
08270931.D	S-N-2 MS	66,600	0.0425	2.8	NA		290%
08270932.D	S-N-2 MSD	69,800	0.0425	3.0	NA		290%

* Ratio of the peak area for L2 to the peak area of L1 expressed as a percentage

RPD = relative percent difference NA = not applicable

The filtrate from each sample was also analyzed for As and Hg by ICP/MS using the analytical method discussed in Section 2.3.7. Samples underwent microwave assisted digestion prior to analysis. The results are shown in Table 31. While the amount of arsenic in the filtrate was

consistent for all solvent types the 7.0 M nitric acid solubilized significantly more mercury from the sludge than water or 20% acetic acid

Table 31 Arsenic and Mercury in Sludge Filtrate

Sample ID	Dissolved Hg (µg/L)	Dissolved As (µg/L)	Hg Sample Mass (µg)	As Sample Mass (µg)
QC-W	<0.5	<0.1	<0.02	<0.004
QC-A	<5.0	6.6	<0.21	0.28
QC-N	<5.0	<1.0	<0.21	<0.04
S-W-1	2,260	348,000	95.9	14,800
S-W-2	1,700	344,000	72.4	14,600
S-A-1	2,880	364,000	122	15,500
S-A-2	2,460	317,000	104	13,500
S-N-1	573,000	359,000	24,400	15,300
S-N-2	577,000	323,000	24,500	13,700

Based on an average sludge starting weight of 0.1 g and using the measured mercury and arsenic concentrations for sludge from sample D-79711-S-01 (see Table 26), the approximate amount of each metal for these tests is

- mercury = 44,000 µg
- arsenic = 15,000 µg

Reviewing the arsenic amounts in each filtrate, it appears that nearly all arsenic was recovered from the sludge for all samples. Much of the mercury for the water and acetic acid samples appears to have remained on the filter, i.e., associated with the undissolved solids, however, greater than 50% of the mercury was removed from the sludge. In an effort to determine a mass balance, the amount of mercury that should have remained on the filter was calculated. Table 32 shows the amount of undissolved solids remaining on each filter and the concentration of mercury in sample D-79711-S-011. From this data the amount of residual mercury was predicted. A complete mass balance cannot be accounted for any of the samples, i.e., the mass of residual mercury on the filter and in the filtrate does not sum to 44,000 µg. About 75% of the mass can be accounted for in nitric acid samples and 30% to 50% can be accounted for in the acetic acid samples. Less than 10% of the mercury can be accounted for in the water samples. Part of the low recovery of mercury may be an artifact related to the low bias for the undissolved solid sample weights discussed above. Additionally, some losses may have occurred during the preparation process where dissolved mercury-containing compounds were not sufficiently transferred from one container to another or through the filtration system.

Table 32. Predicted Mass of Hg Remaining on the Filters

Sample ID	Undissolved Solids (g)	D-79711-S-01 Hg ($\mu\text{g/g}$)	Residual Hg (μg)
S-W-1	0.008	438,000	3,504
S-W-2	0.007	438,000	3,066
S-A-1	0.0296	438,000	12,965
S-A-2	0.0539	438,000	23,608
S-N-1	0.0189	438,000	8,278
S-N-2	0.0185	438,000	8,103

2.5 Lewisite Ton Container Rinse Test

2.5.1 Solubility Test

A preliminary solubility test was performed by placing 3.7 g (1.9 mL) of Lewisite from ton container D-79685 into a 1-L glass bottle. This was done in duplicate. The first bottle, labeled L1, then had 270 mL of 20% acetic acid solution added to the bottle. The second bottle, labeled L2, had 160 mL of de-ionized water added followed by 110 mL of 50% acetic acid to create a 20% acetic acid solution. For both samples, the Lewisite turned from black to brown and adhered to the bottom of the bottle (see Figure 5).

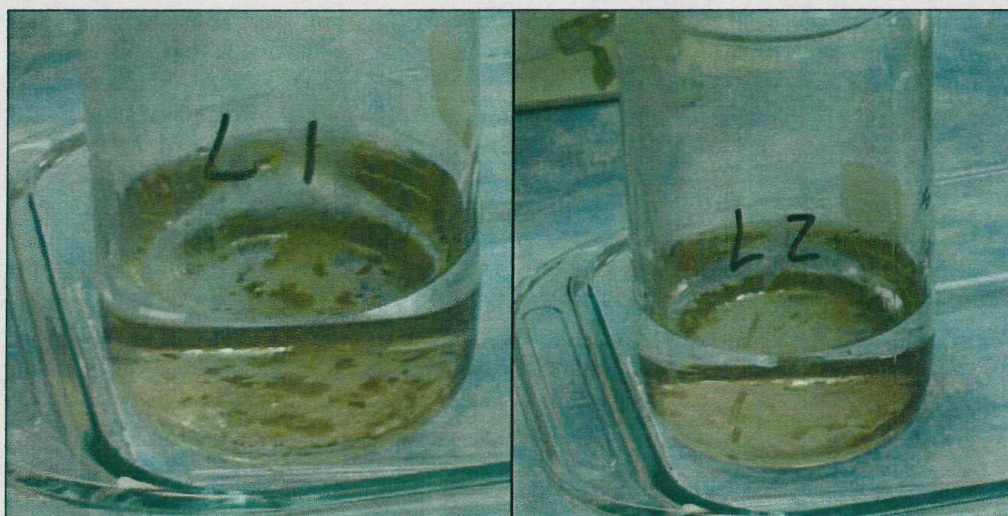


Figure 5. Lewisite with 20% acetic acid.

Bottles were then placed on ball mills and rotated at approximately two revolutions per minute (rpm). Lewisite smeared the sides of the bottles during rotation (see Figure 6). Bottles were allowed to rotate overnight. The following morning (after approximately 20 hr of rotation) the samples did not look appreciably different (see Figure 7) aside from the Lewisite being more evenly distributed on the walls of the bottle.



Figure 6. Bottles L1 (in front) and L2 rotating on ball mills at T=0 hr.

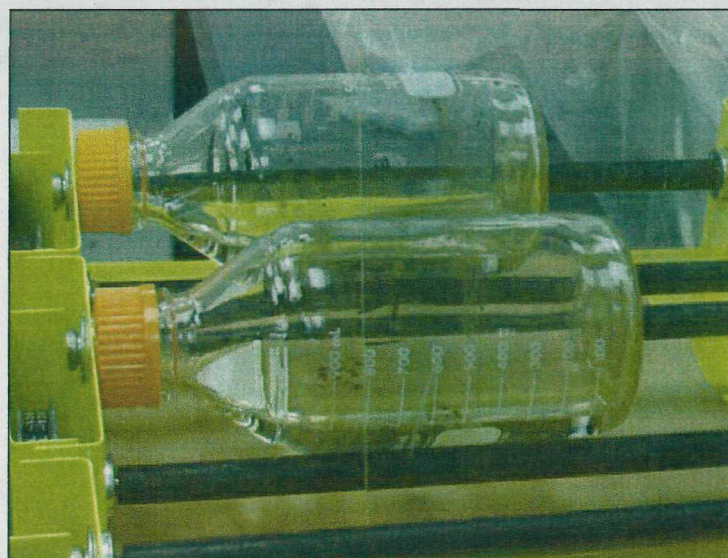


Figure 7. Bottles L1 (in front) and L2 rotating on ball mills at T=20 hr.

The acetic acid from each bottle was then decanted, allowing as much solid material as possible to remain. Ten mL of each acetic acid sample was then returned to the appropriate parent bottle. Next, 150 mL of 7.0 M nitric acid was added to each bottle, swirled to mix and allowed to stand (bottles were not rotated). No obvious change in the bottle contents was observed (see Figure 8).



Figure 8. Samples L1 and L2 after nitric acid addition.

These test appeared to indicate that 20% acetic acid solution could not solubilize Lewisite at the desired ratio of 1:135 (v:v).

2.5.2 Rinse Test

A test to evaluate a procedure for rinsing drained Lewisite ton containers was detailed in Section 3.6.2 of the Test Plan. This test was designed per the Statement of Work contained within the *Request for Proposals for Sample Analysis - CAMDS*. This test was performed in duplicate, however, addition of 20% acetic acid to the test containers was accomplished by two means:

- Rinse Test A – add 1350 mL of 20% acetic acid
- Rinse Test B – add 810 mL of water and then add 540 mL of 50% acetic acid

Duplicate samples were prepared by adding four ton container coupons to each of two 2 L glass bottles (labeled A and B). 10 mL of Lewisite from ton container D-79685 was then added to each bottle with as much Lewisite added on top of the coupons as possible (see Figure 9). These steps (Steps 1 and 2 in the Test Plan Appendix B) were performed on August 21, 2009. Samples were then allowed to sit, loosely capped, until August 25, 2009. This allowed for extended interaction of the Lewisite with the ton container material. After the extended exposure TC coupons were visibly coated with matte black material.

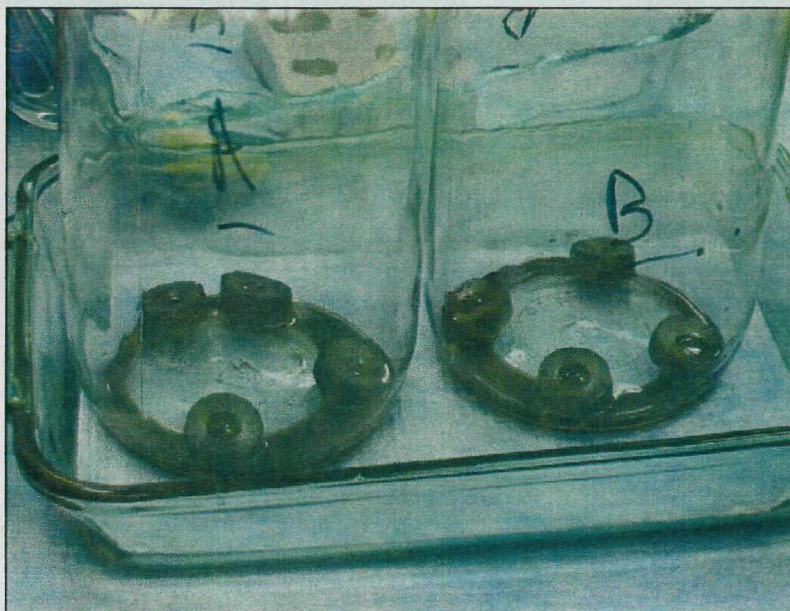


Figure 9. TC coupons with 10 mL of Lewisite.

Two events on August 25, 2009 led to changes in the Test Plan. First, addition of 1350 mL of 20% acetic acid to the Lewisite and ton container coupons per Step 3 in Test Plan Appendix B resulted in a brown, viscous precipitate which coated the bottom and sides of each bottle. These samples were designated as HOAC-1A and HOAC-1B. Only minimal solubilization of the Lewisite by the acetic acid was apparent, similar to what was observed for the Solubility Test described in 2.5.1. Discussion of this result with EG&G led to the decision to skip from Step 5 to Step 35, bypassing additional acetic acid rinsing and going directly to rinsing with 7.0 M nitric acid.

Second, as part of the testing change, one TC coupon and all but 10 mL of 20% acetic acid rinse were removed from the bottles holding samples HOAC-1A and HOAC-1B. 7.0 M nitric acid was then added to the bottle that had contained HOAC-1A to create sample Nitric-3A. As the nitric acid was added, a reaction occurred with the three TC coupons forming a significant amount of NO_x gas and foam. The foam boiled over the edge of the 2-L bottle. See Figure 10 for a photo of the reaction. Only about 600 mL nitric acid was added out of 1350 mL designated for addition. This test was then terminated due to the uncontained exothermic reaction.



Figure 10. Reaction of 7.0 M nitric acid with sample HOAC-1A (left) and after the reaction (right).

Discussion of this result with EG&G led to testing changes captured in a change memo to SOP HMRC X-243-00 and a Test Plan change memo dated September 2, 2009. These changes included:

1. The three TC coupons were removed from sample Nitric-3A and archived. The remaining nitric acid was then added to bottle and the sample archived.
2. Two TC coupons were removed from HOAC-1B and archived. Testing with HOAC-1B then continued with 1350 mL of 7.0 M nitric acid added slowly to the sample bottle, to control the reaction that was evidenced by foaming and NO_x formation. During the nitric acid addition, the one TC coupon was removed to slow the reaction; less than half of the coupon appeared to be remaining. The sample, designated HOAC-1B Nitric, was then allowed to stand overnight.

The following day, 10 mL of HOAC-1B Nitric was added to a new 2-L bottle along with 1350 mL of de-ionized water and spun at 2 rpm for 1 hr. This DI water rinse was repeated 2 more times with 10 mL of the previous rinse cascading to the next rinse. A total of three DI water rinse samples was generated. See Table 33 for a description of each sample.

3. Two new samples were prepared with one TC coupon placed in each of two 2 L bottles and 1.0 mL of Lewisite from ton container D-79685 also placed in each bottle, on top of the coupons. This agent sat in contact with the TC coupons for < 2 hr prior to the addition of nitric acid. One bottle had 135 mL of 3.0 M nitric acid slowly added to create sample L-HNO₃-3. No visible reaction occurred. The second bottle had 135 mL of 7.0 M nitric acid added to create sample L-HNO₃-7. A reaction occurred involving foaming and NO_x formation. Both samples were allowed to stand overnight.

The following day, 10 mL of each sample (L-HNO3-3 and L-HNO3-7) was added to a new 2-L bottle along with the TC coupon from each sample and 1350 mL of de-ionized water and spun at 2 rpm for 1 hr. This DI water rinse was repeated for each sample 2 more times with 10 mL of the previous rinse and TC coupon cascading to the next rinse. A total of three DI water rinse samples was generated for each sample. See Table 33 for a description of each sample.

Table 33 Description of Rinse Test Sample Preparation

Sample ID	Prep Date	Starting Sample Material	Sample Material Vol (mL)	No. of Coupons	Rinse Solution	Rinse Solution Vol (mL)	Rinse Time
HOAC-1A	8/25/2009	Neat Lewisite	10	4	20% Acetic Acid	1350	2 hr
HOAC-1B	8/25/2009	Neat Lewisite	10	4	20% Acetic Acid	1350	2 hr
Nitric-3A	8/25/2009	Residual Material from HOAC-1A	Unknown	3 from HOAC-1A	7.0 M Nitric Acid	~600	NA
HOAC-1B Nitric	8/26/2009	Residual Material from HOAC-1B	Unknown	1 from HOAC-1B	7.0 M Nitric Acid	1350	Stood Overnight
HOAC-1B R1	8/27/2009	HOAC-1B Nitric	10	0	DI Water	1350	1 hr
HOAC-1B R2	8/27/2009	HOAC-1B R1	10	0	DI Water	1350	1 hr
HOAC-1B R3	8/27/2009	HOAC-1B R2	10	0	DI Water	1350	1 hr
L-HNO3-3	8/26/2009	Neat Lewisite	10	1	3.0 M Nitric Acid	135	Stood Overnight
L-HNO3-3 R1	8/27/2009	L-HNO3-3	10	1 from L-HNO3-3	DI Water	1350	1 hr
L-HNO3-3 R2	8/27/2009	L-HNO3-3 R1	10	1 from L-HNO3-3 R1	DI Water	1350	1 hr
L-HNO3-3 R3	8/27/2009	L-HNO3-3 R2	10	1 from L-HNO3-3 R2	DI Water	1350	1 hr
L-HNO3-7	8/26/2009	Neat Lewisite	10	1	7.0 M Nitric Acid	135	Stood Overnight
L-HNO3-7 R1	8/27/2009	L-HNO3-7	10	1 from L-HNO3-7	DI Water	1350	1 hr
L-HNO3-7 R2	8/27/2009	L-HNO3-7 R1	10	1 from L-HNO3-7 R1	DI Water	1350	1 hr
L-HNO3-7 R3	8/27/2009	L-HNO3-7 R2	10	1 from L-HNO3-7 R2	DI Water	1350	1 hr

NA = not applicable - sample reacted violently with nitric acid and test was terminated

Samples were prepared and analyzed by selected ion monitoring GC/MS in accordance with the procedures reported in ECBC-TR-531 for the analysis of L1, L2 and L3. Calibration standards were prepared from Lewisite stock available at the HMRC. A six-point calibration was prepared ranging from 5 ng/mL to 1,000 ng/mL. A method detection limit (MDL) study for L1 was performed per 40 CFR Part 136 Appendix with a resulting MDL value of 24 µg/L. The quantitation limit for a 0.5-mL sample extracted into 5.0 mL of 1% ethane thiol in 2,2,4-trimethylpentane was 50 µg/L. See the Test Plan for method QA/QC criteria.

Results of sample analysis are shown in Table 34. As the Lewisite stock contained primarily L1, quantitative analysis of rinse samples for L2 and L3 utilizing methodology described in ECBC-TR-531 was not possible. L3 was not detected, however, the peak area of L2 was compared to the peak area of L1 in each sample. The results were expressed as a percentage of L2 relative to L1, shown in Table 34. The amount of L1 in HOAC-1A and HOAC-1B was similar, indicating that the approach to adding acetic acid, either as 20% acetic acid or as water followed by 50% acetic acid to create 20% acetic acid, resulted in similar Lewisite solubilization. The higher amount of L1 in the Nitric-3A sample relative to the HOAC-1B Nitric sample may be related to differences in how the two rinses were generated. For the Nitric-3A sample, there were 3 TC coupons present and the reaction was uncontrolled, resulting in excessive foaming and NO_x formation. The HOAC-1B Nitric sample had only 1 TC coupon and the addition of the 7.0 M nitric acid was more controlled. Also, the duration of the nitric acid rinse was overnight. The reaction that occurred with the Nitric-3A sample may have resulted in a rapid reduction in the effectiveness of the nitric acid for decomposing L1, as the acid may have primarily reacted with the TC coupon material. Alternately, the longer exposure time (overnight) of the HOAC-1B Nitric sample to the 7.0 M nitric acid may have resulted in greater L1 decomposition.

The rinse tests that began with nitric acid (samples L-HNO₃-3 and L-HNO₃-7) appeared to result in lower L1 in the nitric acid rinsate than the samples that began with acetic acid (Nitric-3A and HOAC-1B Nitric). The reason for this is not immediately apparent. L1 was not detected in any of the DI water rinses, as might be expected considering that a 1:135 dilution of the starting nitric acid rinsates would result in L1 concentrations below the method quantitation level.

The response of L2 compared to L1 for samples HOAC-1A, HOAC-1B and Nitric 3A was close to native levels in Lewisite (~15%). The samples HOAC-1B Nitric, L-HNO₃-3 and L-HNO₃-7 had higher levels of L2. This difference may be due to either better solubilization of L2 for those tests or less decomposition of L2 relative to L1. Sample HOAC-1B R1 did not contain L1 but did contain L2. While a relative ratio could not be determined for this sample, the percentage of L2 relative to the amount of L2 found in the HOAC-1B Nitric sample (0.99%) was consistent with a 1:135 dilution of HOAC-1B Nitric.

Table 34 L1 and L2 in Rinse Samples

Sample ID	Data File Name	L1 Conc (µg/L)	Area L2 / Area L1*
HOAC-1A	08260914 D	800,000	15%
HOAC-1B	08260916 D	920 000	18%
Nitric-3A	08260920 D	61,000	24%
HOAC-1B Nitric	08280913 D	4,800	261%
HOAC-1B R1	08280912 D	<50	NC
HOAC-1B R2	08270943 D	<50	ND
HOAC-1B R3	08270937 D	<50	ND
L-HNO3-3	08270954 D	980	107%
L-HNO3-3 R1	08270947 D	<50	ND
L-HNO3-3 R2	08270944 D	<50	ND
L-HNO3-3 R3	08270938 D	<50	ND
L-HNO3-7	08270952 D	1 600	37%
L-HNO3-7 R1	08270951 D	<50	ND
L-HNO3-7 R2	08270945 D	<50	ND
L-HNO3-7 R3	08270939 D	<50	ND

* Ratio of the peak area for L2 to the peak area of L1 expressed as a percentage

NC = not calculated – L2 detected but L1 not detected

ND = L2 not detected

Arsenic and mercury in the rinse samples were determined by ICP-MS following SOP HMRC X-241. Samples underwent microwave assisted digestion prior to analysis. Based on the mercury and arsenic concentrations in sample D-79685-L the nominal mercury concentration (assuming 100% solubilization in the first rinse) would be 7,600 µg/L and the nominal arsenic concentration would be 4,600,000 µg/L. This is based on a starting volume of either 10 mL for 1.0 mL of lewisite and an initial rinse volume of either 1350 mL or 135 mL. Reviewing the mercury and arsenic rinse test results shown in Table 35, in particular for samples L-HNO3-3 and L-HNO3-7, it appears that both 3.0 M and 7.0 M nitric acid are adequate for solubilizing a majority of mercury and arsenic. The results for samples HOAC-1A and HOAC-1B indicate that 20% acetic acid is not as effective for solubilizing mercury and arsenic. Comparing the concentration of arsenic in samples HOAC-1A and HOAC-1B to the concentration of L1 in the same samples, the ratio as a percentage (55% and 49% respectively) is similar to the mass percent of arsenic in L1 (37%). The ratios are even closer to 37% if the abundance of L2 is also taken into account. This similarity indicates that a large proportion of the As exists in the acetic acid rinse as Lewisite with very little L1 decomposition occurring. Conversely, the very high concentrations of arsenic in samples L-HNO3-3 and L-HNO3-7 compared to the concentrations of L1 in the same samples indicates that Lewisite decomposition has occurred during these rinses. The DI water rinses for samples L-HNO3-3 and L-HNO3-7 resulted in concentrations of both mercury and arsenic consistent with 1:135 sample dilutions, as would be expected.

Table 35 Hg and As in Rinse Samples

Sample ID	Hg (µg/L)	As (µg/L)
HOAC-1A	665	438,000
HOAC-1B	502	449,000
Nitric-3A	10 400	1,070,000
HOAC-1B Nitric	2,220	159,000
HOAC-1B R1	5 64	2,650
HOAC-1B R2	<5 00	28 7
HOAC-1B R3	<5 00	31 8
L-HNO3-3	4 680	5,080,000
L-HNO3-3 R1	51 6	37 300
L-HNO3-3 R2	<5 00	334
L-HNO3-3 R3	<5 00	132
L-HNO3-7	6,794	5,650,000
L-HNO3-7 R1	61 3	42,100
L-HNO3-7 R2	<5 00	259
L-HNO3-7 R3	<5 00	57 5

The metal coupons were analyzed for residual Lewisite using the method described in ECBC-TR-531. All sample coupons were extracted in a single batch. Two coupon blanks and two spiked coupons were extracted and analyzed. Coupons were extracted for 15 min with 25 mL of 2,2,4-trimethylpentane containing 1% ethanethiol by volume. Coupon extracts were analyzed in the same manner as the liquid samples. Results for L1 were reported as µg/coupon. L3 was not detected. The peak area of L2 was compared to the peak area of L1 in each sample. This was expressed as a percentage of L2 relative to L1. Concentrations of L1 were expressed as µg/coupon and are shown in Table 36. Samples COUPON HOAC-B1 and COUPON HOAC-B3 are replicates taken from the same parent rinse sample, HOAC-1B. The acetic acid rinses of the coupons left residual L1 on the coupons, however, the nitric acid rinses (with and without DI water rinses) reduced the concentration of L1 by one to two orders of magnitude. The amounts of L2 relative to L1 remaining on the TC coupons were fairly high in all cases, indicating that L2 may be able to better sorb to TC metal surfaces than L1. This result also indicates that the higher levels of L2 relative to L1 in the rinse samples is due to better decomposition of L1 as opposed to better solubilization of L2. The MS and MSD samples resulted in good recoveries of 56% and 53%, respectively. No L1 or L2 was detected in the blanks.

Table 36 L1 and L2 on Ton Container Coupons

Coupon Sample ID	Data File Name	Associated Sample	L1 Conc (µg/coupon)	Area L2 / Area L1*
COUPON HOAC-A1	08310914 D	HOAC-1A	120	846%
COUPON HOAC-B1	08310913 D	HOAC-1B	240	239%
COUPON HOAC-B3	08310912 D	HOAC-1B	140	568%
COUPON HNO3-A1	08280921 D	Nitric-3A	8.5	550%
COUPON L-HNO3-3	08280920 D	L-HNO3-3 R3	3.4	140%
COUPON L-HNO3-7	08280916 D	L-HNO3-7 R3	0.87	486%

* Ratio of the peak area for L2 to the peak area of L1 expressed as a percentage

Following extraction for residual Lewisite, recoverable arsenic and mercury on the same TC coupons was determined using ICP-MS analysis after extraction with 25 mL of 0.1 N HNO₃. Samples were heated to ~50°C for 30 min to enhance extraction and then analyzed. All sample coupons were extracted in a single batch along with two blank coupons and two spiked coupons. Results for As and Hg were reported as µg/coupon in Table 37. The amount of residual mercury found on each coupon was inconsistent, the RPD for the mercury in the duplicate samples was 170%. This could be due to varying interaction between the mercury and the TC coupon surface. The amount of arsenic for each coupon was much higher than would be expected based on L1 concentrations. The arsenic levels could be related to the presence of L2 or arsenic trichloride, which may interact more strongly with the TC coupon surface than L1.

Table 37 Hg and As on Ton Container Coupons

Coupon Sample ID	Associated Sample	Hg (µg/coupon)	As (µg/coupon)
Coupon Blank 1	NA	<0.13	<0.025
Coupon Blank 2	NA	<0.13	<0.025
COUPON HOAC-A1	HOAC-1A	0.54	1,300
COUPON HOAC-B1	HOAC-1B	6.15	3,600
COUPON HOAC-B3	HOAC-1B	0.58	2,950
COUPON HNO3-A1	Nitric-3A	0.69	35.1
COUPON HNO3-3	L-HNO3-3 R3	2.72	58.8
COUPON HNO3-7	L-HNO3-7 R3	0.67	10.3

2.5.3 Reaction Calorimetry Tests

Solution calorimetry was performed to obtain heat of reaction/dissolution data and to calculate pressure data when Lewisite was mixed with a 3.0 M nitric acid solution and when Lewisite solution was mixed with a 20% acetic acid solution. The testing was performed using a Mettler RC1e reaction calorimeter with an 80-mL reaction vessel. A 1-L Tedlar® bag was connected to the reactor to collect any gas evolved during the reaction in order to calculate an increase in pressure.

In the first calorimetry test, the 80 mL reactor was loaded with 58.0 mL of 3.0 M nitric acid. The reactor temperature (T_r) was brought to 24.0°C and allowed to equilibrate and calibrate at this temperature. Once the calibration was completed, 450 μ L (0.88 g) of Lewisite from ton container D-79685 was added to the reactor in a drop-wise fashion over one minute using a gas-tight syringe with a 4-inch needle to ensure that the agent was added directly to the acid. The propeller speed was set at 200 rpm. When added, the agent dropped to the bottom of the reactor. The propeller speed was increased to 400 rpm to better mix the agent, some agent was dispersed into the solution but a majority swirled at the bottom of the reactor. The heat of reaction was measured in a small window of time around the point of agent addition. In the case of the 3.0 M nitric acid, the reaction was measured over a 4-minute period, no visible change was observed in the agent at the end of 4 minutes. The reaction was exothermic, generating 0.0779 kJ of heat energy. The adiabatic temperature rise was 0.323 K. After 1 hr, the T_r was increased to 50.0°C to see if the Lewisite would go into solution. The increase in temperature did not visibly further solubilize the Lewisite, the agent continued to swirl at the bottom of the reactor, no additional heat data were recorded. The exotherm generated was minimal. No evolved gas was collected in the Tedlar bag during the test, therefore, no reaction pressure increase was calculated. See Table 38 for a summary of results, including the heat generated per mass of agent as well as the specific heat of the reaction determined at the end of the trial.

In the second calorimetry test, the 80 mL reactor was loaded with 60.0 mL of 20% Acetic Acid. The T_r was brought to 24.0°C and allowed to equilibrate and calibrate at that temperature. Once the calibration was completed, 450 μ L (0.88 g) of Lewisite from ton container D-79685 was added to the reactor in a drop-wise fashion over one minute using a gas-tight syringe with a 4-inch needle to ensure that the agent went directly into the acid. The reactor temperature was observed to increase during the addition. When added, the agent dropped to the bottom of the reactor. This time, the propeller speed was already at 400 rpm. As before, some of the agent was dispersed into the solution but a majority swirled at the bottom of the reactor. The heat of reaction was measured in a small window of time around the point of agent addition. In the case of the 20% nitric acid, the reaction was measured over a 6-minute period. No visible change was observed in the agent at the end of 6 minutes, the agent continued to swirl at the bottom of the reactor. The reaction was exothermic, generating 0.129 kJ of heat energy. The adiabatic temperature rise was 0.503 K. After 1 hr the T_r was increased to 50.0°C to see if the Lewisite would go into solution. The increase in temperature did not visibly further solubilize the Lewisite. The exotherm generated was minimal. Sufficient gas did not evolve during the experiment to fill the Tedlar bag, therefore, no reaction pressure increase was calculated. See Table 38 for a summary of results, including the heat generated per mass of agent as well as the specific heat of the reaction determined at the end of the trial.

Table 38 Lewisite Calorimetry Results

Trial Number	Reactant	Agent Mass (g)	Adiabatic ΔT (K)	Heat of Reaction (kJ)	Heat/Agent Mass (kJ/g)	Specific Heat: [kJ / (kg·K)]
nitnCL8-27-09	3.0 M Nitric Acid	0.88	0.327	0.0795	0.0903	3.64
AAL8-31-09	20% Acetic Acid	0.88	0.503	0.129	0.147	3.86

3 0 REFERENCES

- 1 "The Sources, Fate, and Toxicity of Chemical Warfare Agent Degradation Products", Munro et al , Volume 107, Number 12, December 1999, Environmental Health Perspectives
- 2 Executive Summary of "Chemical Warfare Agents and their Hydrolysis Products from the US EPA Standardized Analytical Methods and GC-MS Analytical Method for the Analysis of Chemical Warfare Agent Degradation Products Listed in the EPA Standardized Analytical Methods", Haigh, Theodore A , Prepared for NEMC Conference, Cambridge, MA, Aug 20-24, 2007
- 3 Request for Proposal for Sample Analysis – CAMDS, JK-P-001, April 3, 2009, EG&G Defense Materials, Inc
- 4 TOCDF LOP TE-LOP-557, Analysis of Metals by ICP-MS
- 5 SW-846 Method 9045D, Soil and Waste pH
- 6 SOP HMRC I-023-15, "Chemical Agent (CA) Receipt, Storage, Accountability and Reporting at the HMRC"
- 7 HMRC IV-055-06, "Purity and Impurity Analysis of Solutions Containing Chemical Agent by Gas Chromatography"
- 8 HMRC IV-056-12, "Operation and Maintenance of Gas Chromatography and Analysis of Solutions Containing GA, GB, GD, GF, HD, L, and VX by Gas Chromatography"
- 9 HMRC IV-067-05, "Quantitative Analysis of HD, GA, GB, GD, and VX Collected on Solid Sorbent"
- 10 HMRC X-0241-00, "Microwave Assisted Digestion of GA and L for HRA Metals Analysis by ICP-MS"
- 11 HMRC X-0243-00, "Ton Container Rinse Test, pH Measurement of L and GA and Solubility of L Sludge"
- 12 HMRC X-0244-00, "Lewisite Agent Hydrolysis Using the Mettler RC1 for Use in the Ton Container Demil Program"
- 13 ECBC-TR-531 Appendix - Standard Operating Procedure for "Multi-Residue Quantitative Analysis of HD, HN3, Lewisite and Other Arsenical Chemical Warfare Agents in Permanganate-Based Demilitarization Waste Streams"
- 14 Battelle Chemical Environmental and Materials Operations (CEMO) Quality Manual

Appendix A

Sample Information

MAIL COURIER RECEIPT		SHIPPER'S CONTROL/DOCUMENT NO W90BDL-09205-3001		PRIVACY ACT STATEMENT				
SHIPPER DCD, Stockton, Utah 84071, PH (435)-833-6096		SUPPLY ACCOUNT NUMBER		AUTHORITY 5 U.S.C. Sec 552a (PL 93-578) PRINCIPLE PURPOSES To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receiving for the material. ROUTINE USES To document transfer of material from a shipper to a courier, courier to courier and/or receiver. DISCLOSURE IS VOLUNTARY. Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.				
DESTINATION Battelle Mem Institute, West Jefferson, Ohio 43162		SUPPLY ACCOUNT NUMBER						
I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.				SHIPMENT DESCRIPTION				
				LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS	
SHIPMENT TRANSFERS				1	1	D-79685-L-01	(1) - 4 mL Lewisite liquid sample from TC D79685	
FIRST	LOCATION OF TRANSFER <i>Bldg 1432</i> <i>Deseret Chemical Depot</i>		DATE (YR/MO/DAY) <i>09/07/24</i>		2	1	D-79685-L-02	(1) -10 mL Lewisite liquid sample from TC D79685
RECIPIENT'S PRINTED NAME (LAST FIRST MI) <i>Shields Kent E.</i>			ORGAN OR ACCOUNT NO <i>Inv. DCD</i>		3	1	D-79685-L-03	(1) -10 mL Lewisite liquid sample from TC D79685
SIGNATURE <i>Kent E. Shields</i>			SOCIAL SECURITY NUMBER <i>[REDACTED]</i>		4	1	D-79685-L-04	(1) -10 mL Lewisite liquid sample from TC D79685
SECOND	LOCATION OF TRANSFER <i>Helipad DCD</i>		DATE (YR/MO/DAY) <i>09/07/24</i>		5	1	D-79699-L-01	(1) - 4 mL Lewisite liquid sample from TC D79699
RECIPIENT'S PRINTED NAME (LAST FIRST MI) <i>Armstrong Richard L</i>			ORGAN OR ACCOUNT NO <i>CANA - EAST</i>		6	1	D-79700-L-01	(1) - 4 mL Lewisite liquid sample from TC D79700
SIGNATURE <i>Richard Armstrong</i>			SOCIAL SECURITY NUMBER <i>[REDACTED]</i>		7	1	D-79701-L-01	(1) - 4 mL Lewisite liquid sample from TC D79701
THIRD	LOCATION OF TRANSFER <i>Battelle West Jefferson, Ohio</i>		DATE (YR/MO/DAY) <i>09/07/24</i>		8	1	D-79703-L-01	(1) - 4 mL Lewisite liquid sample from TC D79703
RECIPIENT'S PRINTED NAME (LAST FIRST MI) <i>CAMPBELL, BRUCE M</i>			ORGAN OR ACCOUNT NO <i>HARC</i>		9	1	D-79705-L-01	(1) - 4 mL Lewisite liquid sample from TC D79705
SIGNATURE <i>Bruce Campbell</i>			SOCIAL SECURITY NUMBER <i>[REDACTED]</i>		10	1	D-79693-L-01	(1) - 4 mL Lewisite liquid sample from TC D79693
FOURTH	LOCATION OF TRANSFER 		DATE (YR/MO/DAY) 		11	1	D-79711-L-01	(1) - 4 mL Lewisite liquid sample from TC D79711
RECIPIENT'S PRINTED NAME (LAST FIRST MI) 			ORGAN OR ACCOUNT NO 		12	1	D-79697-L-01	(1) - 4 mL Lewisite liquid sample from TC D79697
SIGNATURE 			SOCIAL SECURITY NUMBER 		13	1	D-79699-S-01	(1) - 2 mL Lewisite solid sample from TC D79699
FIFTH	LOCATION OF TRANSFER 		DATE (YR/MO/DAY) 		14	1	D-79700-S-01	(1) - 2 mL Lewisite solid sample from TC D79700
RECIPIENT'S PRINTED NAME (LAST FIRST MI) 			ORGAN OR ACCOUNT NO 		15	1	D-79701-S-01	(1) - 2 mL Lewisite solid sample from TC D79701
SIGNATURE 			SOCIAL SECURITY NUMBER 		16	1	D-79703-S-01	(1) - 2 mL Lewisite solid sample from TC D79703

MA COURIER RECEIPT			SHIPPER'S CONTROL/DOCUMENT NO W90BDL-09205-3001		PRIVACY ACT STATEMENT	
SHIPPER DCD, Stockton, Utah 84071, PH (435) 833-6096			SUPPLY ACCOUNT NUMBER		AUTHORITY 5 U.S.C. Sec 552a (PL 93 579) PRINCIPLE PURPOSES To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receiving for the material. ROUTINE USES To document transfer of material from a shipper to a courier, courier to courier and/or receiver. DISCLOSURE IS VOLUNTARY. Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.	
DESTINATION Battelle Mem Institute, West Jefferson, Ohio 43162			SUPPLY ACCOUNT NUMBER			
I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.					SHIPMENT DESCRIPTION	
					LINE NUMBER	QUANTITY
SHIPMENT TRANSFERS					SERIAL NUMBERS	REMARKS
FIRST LOCATION OF TRANSFER 1432 Bldg Desert Chemical Depot DATE (YR/MO/DAY) 09/07/24					17	1
					D-79705-S-01	(1) - 2 mL Lewisite solid sample from TC D79705
RECIPIENT'S PRINTED NAME (LAST FIRST MI) Shields Kent E ORGAN OR ACCOUNT NO DCD Inventory SIGNATURE Kent E Shields SOCIAL SECURITY NUMBER					18	1
					D-79685-S-01	(1) - 2 mL Lewisite solid sample from TC D79705
SECOND LOCATION OF TRANSFER Helipad DCD DATE (YR/MO/DAY) 09/07/24					19	1
					D-49221-L-01	(1) - 4 mL Lewisite liquid sample from TC D49221
RECIPIENT'S PRINTED NAME (LAST FIRST MI) Armstrong Richard L ORGAN OR ACCOUNT NO CARA-FAST SIGNATURE Richard Armstrong SOCIAL SECURITY NUMBER					20	1
					D-49221-L-D	(1) - 4 mL Lewisite liquid sample from TC D49221
THIRD LOCATION OF TRANSFER Battelle West Jefferson, Ohio DATE (YR/MO/DAY) 09/07/24					21	1
					D-49221-S-01	(1) - 2 mL Lewisite solid sample from TC D49221
RECIPIENT'S PRINTED NAME (LAST FIRST MI) CAMPBELL, BRUCE M ORGAN OR ACCOUNT NO HARC SIGNATURE Bruce Campbell SOCIAL SECURITY NUMBER					22	1
					D-79693-S-01	(1) - 2 mL Lewisite solid sample from TC D79693
FOURTH LOCATION OF TRANSFER DATE (YR/MO/DAY)					23	1
					D-79697-S-01	(1) - 2 mL Lewisite solid sample from TC D79697
FIFTH LOCATION OF TRANSFER DATE (YR/MO/DAY)					24	1
					D-79711-S-01	(1) - 2 mL Lewisite solid sample from TC D79711
RECIPIENT'S PRINTED NAME (LAST FIRST MI) CAMPBELL, BRUCE M ORGAN OR ACCOUNT NO HARC SIGNATURE Bruce Campbell SOCIAL SECURITY NUMBER					//////////	//////////LAST ITEM////////
RECIPIENT'S PRINTED NAME (LAST FIRST MI) ORGAN OR ACCOUNT NO SIGNATURE SOCIAL SECURITY NUMBER						
RECIPIENT'S PRINTED NAME (LAST FIRST MI) ORGAN OR ACCOUNT NO SIGNATURE SOCIAL SECURITY NUMBER						
RECIPIENT'S PRINTED NAME (LAST FIRST MI) ORGAN OR ACCOUNT NO SIGNATURE SOCIAL SECURITY NUMBER						

MAIL COURIER RECEIPT		SHIPPER'S CONTROL/DOCUMENT NO W90BDL-09205-3002/3003		PRIVACY ACT STATEMENT			
SHIPPER DCD, Stockton Utah 84071, PH (435) 833-6096		SUPPLY ACCOUNT NUMBER		AUTHORITY 5 U.S.C. Sec 552a (PL 93 579) PRINCIPLE PURPOSES To provide a receipt for transfer of controlled material. The use of the SSAN is required and is necessary to provide positive identification of the individuals receiving for the material. ROUTINE USES To document transfer of material from a shipper to a courier, courier to courier and/or receiver. DISCLOSURE IS VOLUNTARY. Since the SSAN must be used, refusal to provide SSAN may be grounds for action to remove the individual concerned from duties involving the material transferred by use of this form.			
DESTINATION Battelle Mem Institute, West Jefferson, Ohio 43162		SUPPLY ACCOUNT NUMBER					
I certify by my signature that I have received the material listed on this form and am aware of the applicable safety and security requirements.				SHIPMENT DESCRIPTION			
				LINE NUMBER	QUANTITY	SERIAL NUMBERS	REMARKS
SHIPMENT TRANSFERS				1	1	D 25253-L 01	(1) - 4 mL GA liquid sample from TC D25253
FIRST	LOCATION OF TRANSFER Bldg. 1732 Deseret Chemical Dept	DATE (YR/MO/DAY) 09/07/24		2	1	D-25253-L-D	(1) - 4 mL GA liquid sample from TC D25253
RECIPIENT'S PRINTED NAME (LAST FIRST MI) Shields Kent E.		ORGAN OR ACCOUNT NO DCD Inventory		3	1	D-35248-L-01	(1) - 4 mL GA liquid sample from TC D35248
SIGNATURE Kent E. Shields		SOCIAL SECURITY NUMBER		4	1	D-29813-L-01	(1) - 4 mL GA/UCON liquid sample from TC D29813
SECOND	LOCATION OF TRANSFER Helipad DCD	DATE (YR/MO/DAY) 09/07/24		5	1	D-51365-L-01	(1) - 4 mL GA/UCON liquid sample from TC D51365
RECIPIENT'S PRINTED NAME (LAST FIRST MI) McIntosh Richard L		ORGAN OR ACCOUNT NO CARA-EAST		//////////		//////////LAST ITEM////////	NOTHING FOLLOWS//////////
SIGNATURE Richard L McIntosh		SOCIAL SECURITY NUMBER					
THIRD	LOCATION OF TRANSFER Battelle West Jefferson, Ohio	DATE (YR/MO/DAY) 09/07/24					
RECIPIENT'S PRINTED NAME (LAST FIRST MI) CAMPBELL, BRUCE M		ORGAN OR ACCOUNT NO HMRC					
SIGNATURE Bruce Campbell		SOCIAL SECURITY NUMBER					
FOURTH	LOCATION OF TRANSFER	DATE (YR/MO/DAY)					
RECIPIENT'S PRINTED NAME (LAST FIRST MI)		ORGAN OR ACCOUNT NO					
SIGNATURE		SOCIAL SECURITY NUMBER					
FIFTH	LOCATION OF TRANSFER	DATE (YR/MO/DAY)					
RECIPIENT'S PRINTED NAME (LAST FIRST MI)		ORGAN OR ACCOUNT NO					
SIGNATURE		SOCIAL SECURITY NUMBER					

UNCLASSIFIED OPSEC SENSITIVE

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80										1 TOTAL PRICE		2 SHIP FROM		3 SHIP TO					
1 D O C U M E N T 2 FROM 3 U I N S I T 4 Q U A N T I T Y 5 S E R I E S 6 S U P P L I E R 7 M E N T A R Y 8 A D D R E S S 9 S I G N 10 I N 11 C O N T A I N E R 12 P R O J E C T 13 P R I 14 R E A D Y 15 A D V 16 R 17 C I G 18 I N 19 D 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80										UNIT PRICE DOLLARS CTS		Deseret Chemical Depot Utah		Battelle Memorial Institute, Ohio					
ASE ATG ML 12ML ATB 1 D										4 MARK FOR									
Document Number & Suffix W90BDL-09205-3002 July 24, 2009										5 DOC DATE		6 NMFC		7 FRT RATE		8 TYPE CARGO		9 PS	
										10 QTY REC'D		11 UP		12 UNIT WEIGHT		13 UNIT CUBE		14 UFC	
National Stock No & Add (8-22) 1365-01-X89-0020										12ML									
										16 FREIGHT CLASSIFICATION NOMENCLATURE									
RIC ATB UI ML Qty 12ML Con Code D Lot # UNKNOWN										17 ITEM NOMENCLATURE									
										GA Agent Sample Not Applicable									
Movement of GA Agent Sample from Deseret Chemical Depot to Battelle Memorial Institute, West Jefferson, Ohio for agent Characterization LSC# 94C19 Samples = D-25253-L-01 = 4ML, D-25253-L-D = 4ML, D-35248-L-01 = 4ML										18 TY CONT		19 NO CONT		20 TOTAL WEIGHT		21 TOTAL CUBE			
										LSC		1				0			
22 RECEIVED BY Bruce Campbell										23 DATE RECEIVED		7/24/09							

PREVIOUS EDITION MAY BE USED

PerFORM (DLA)

Truck 315A
Seal 09175

UNCLASSIFIED OPSEC SENSITIVE

Becky S Davis
24 Jul 09

UNCLASSIFIED OPSEC SENSITIVE

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80										1 TOTAL PRICE		2. SHIP FROM		BATTELLE MEMORIAL INSTITUTE OHIO					
DESERET CHEMICAL DEPOT UTAH										4 MARK FOR									
UNIT PRICE										DOLLARS		CTG							
DOLLARS										CTG									
5 DOC DATE										6 NMFC		7 FRT RATE		8 TYPE CARGO		9. PS			
10. OTY REC'D										11 UP		12. UNIT WEIGHT		13. UNIT CUBE		14. UPC		15. SL	
16. FREIGHT CLASSIFICATION NOMENCLATURE																			
17 ITEM NOMENCLATURE																			
L Agent Sample Not Applicable Not Applicable																			
18 TY CONT										19 NO CONT		20 TOTAL WEIGHT		21 TOTAL CUBE					
MRC										2				0					
22. RECEIVED BY																23. DATE RECEIVED			
Bene Cepbell																7/24/09			

Document Number & Suffix: W90BDL-09205-3001 24 JULY 2009

National Stock No & Add (8-22): 1365 01 X780021
89
mc 7/27/09

RIC: ATB
 UI: ML
 Qty: TOTAL 94 mL
 Con Code: D
 Lot #: 60 SEE BELOW FOR SN

1 TOTAL PRICE

2. SHIP FROM

DESERET CHEMICAL DEPOT UTAH

4 MARK FOR

UNIT PRICE

DOLLARS

CTG

5 DOC DATE

6 NMFC

7 FRT RATE

8 TYPE CARGO

9. PS

10. OTY REC'D

11 UP

12. UNIT WEIGHT

13. UNIT CUBE

14. UPC

15. SL

16. FREIGHT CLASSIFICATION NOMENCLATURE

17 ITEM NOMENCLATURE

L Agent Sample Not Applicable Not Applicable

18 TY CONT

19 NO CONT

20 TOTAL WEIGHT

21 TOTAL CUBE

MRC

2

22. RECEIVED BY

Bene Cepbell

23. DATE RECEIVED

7/24/09

Movement of Lewisite Chemical Agent Samples from Deseret Chemical Depot, Stockton Utah to Battelle Memorial Institute West Jefferson, Ohio
 24 samples are packaged into 2 MRC (containers)

#1 MRC JJ-212-PB D 79685-L-01 (4mL) D-79685-L-02 (10mL) D-79685-L-03 (10mL) D-79685-L-04 (10mL) D-79699-L 01 (4mL) D-79700-L-01 (4mL) D-79701-L 01 (4mL), D-79703-L-01 (4mL) D-79705-L-01 (4mL) D-79693-L-01 (4mL) D-79711-L-01 (4mL) D-79697-L-01 (4mL), D-79699-S-01 (2mL) D-79700-S-01 (2mL) D-79701-S-01 (2mL) D-79703-S-01 (2mL) D-79705-S-01 (2mL), D-79685-S-01 (2mL)

#2 MRC JJ-079-PB D-49221-S-01 (2mL), D-79693-S-01 (2mL) D-79697-S-01 (2mL) D-79711-S-01 (2mL) D-49221-L-01 (4mL), D-49221-L-D (4mL)

PREVIOUS EDITION MAY BE USED

PERFORM (DLA)

Truck 315A
Seq 09175

UNCLASSIFIED OPSEC SENSITIVE

Becky & Davis
24 Jul 09

Ton Container Sampling Log Form						
Intrusive Sampling of the GA Ton Container Inventory for TOCDF						
Ton Container Number Example D-XXXXX	Sample ID Number Example D-XXXX-M-NN	Collection Ordinal Date/Time Example 2009001- 0800	Sample Volume Example 2 mL	Sample Taken by	Received Sample	Date
	D-25253-S-01		2.0 mL	N/A		
	D-25253-L-01	D25253-L-01	4.0 mL	Jennings, Donald <i>[Signature]</i>	<i>Kate E. Shuler</i>	07/15/09
D-25253				Received for analysis	<i>[Signature]</i>	07/24/09
	D-25253-L-B	D-25253-L-D	4.0 mL	Jennings, Donald <i>[Signature]</i>	<i>Kate E. Shuler</i>	07/15/09
				Received for analysis	<i>[Signature]</i>	07/24/09

D-35248	D-35248-S-01		2.0 mL	N/A		
	D-35248-L-01	D-35248-L-01	4.0 mL	Noel, Anthony <i>Anthony Noel</i>	<i>Yvette Schuch</i>	07/15/09
				Received for analysis	<i>Robert Hunter</i>	07/24/09
D-29813					<i>Stacy Wilgus</i>	07/24/09
	D-29813-S-01		2.0 mL	N/A		
	D-29813-L-01	D-29813-L-01	4.0 mL	Jennings, Donald <i>Donald Jennings</i>	<i>Yvette Schuch</i>	07/16/09
				Received for analysis	<i>Robert Hunter</i>	07/24/09
					<i>Stacy Wilgus</i>	07/24/09

20:00

20:00

D-51365	D-51365-S-01		20 mL	N/A		
	D-51365 L-01	D-51365-L-01	40 mL	<small>Moel, Anthony</small> <i>Anthony Moel</i>	<i>Kent G. Schaefer</i>	07/16/09
				<i>Received for analysis</i>	<i>Robert Hunter</i>	07/24/09
					<i>John G. McKee</i>	07/24/09

20:00

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewistown Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Taken by	Received Sample	Date
D-49221	D-49221-S-01		2.0 mL	Noel, Anthony <i>Anthony Noel</i>	<i>Kent E. Schuch</i>	07/09/09
					<i>Rachel H. Hester</i>	07/24/09
				Received for analysis.	<i>John L. Wilcox</i>	07/24/09
	D-49221-L-01		4.0 mL	Noel, Anthony <i>Anthony Noel</i>	<i>Kent E. Schuch</i>	07/09/09
					<i>Rachel H. Hester</i>	07/24/09
				Received for analysis	<i>John L. Wilcox</i>	07/24/09
	D-49221-L-B		4.0 mL	Noel, Anthony <i>Anthony Noel</i>	<i>Kent E. Schuch</i>	07/09/09
					<i>Rachel H. Hester</i>	07/24/09
				Received for analysis	<i>John L. Wilcox</i>	07/24/09

20:08

20:00

20:00

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID#; M = matrix type (S = sludge L = liquid G = sorbent tube); NN = sequential # (01 02, etc.) or B = Blank.

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewisite Ton Container Inventory for TOCDF

Ton Container Number Example: D-XXXXX	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Taken by	Received Sample	Date
D-79685	D-79685-S-01		2.0 mL	Jennings, Donald	Kent E. Schaefer	07/09/09
					Robert Hunter	07/24/09
				Received for analysis	Kurt W. Wyle	07/24/09
	D-79685-L-01		4.0 mL	Jennings, Donald	Kent E. Schaefer	07/09/09
					Robert Hunter	07/24/09
				Received for analysis	Kurt W. Wyle	07/24/09
	D-79685-L-02		10.0 mL	Jennings, Donald	Kent E. Schaefer	07/09/09
	D-79685-L-03		10.0 mL	Jennings, Donald	Kent E. Schaefer	07/09/09
	D-79685-L-04		10.0 mL	Jennings, Donald	Kent E. Schaefer	07/09/09
					Robert Hunter	07/24/09
				Received for analysis	Kurt W. Wyle	07/24/09

20:00

20:00

20:00

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID#, M = matrix type (S = sludge, L = liquid, G = sorbent tube) NN = sequential # (01, 02, etc.) or B = Blank.

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewisite Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Taken by	Received Sample	Date
D-79693	D-79693-S 01		2.0 mL	<i>Donald Jennings</i>	<i>Kate Schuler</i>	07/10/09
				<i>Received for</i>	<i>Richard Hunter</i>	07/24/09
				<i>analysis</i>	<i>Fluor. Mgr</i>	07/24/09 20:00
	D-79693-L-01		4.0 mL	<i>Donald Jennings</i>	<i>Kate Schuler</i>	07/10/09
				<i>Received for</i>	<i>Richard Hunter</i>	07/24/09
				<i>analysis</i>	<i>Fluor. Mgr</i>	07/24/09 20:00

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID# M = matrix type (S = sludge, L = liquid, G = sorbent tube) NN = sequential # (01, 02, etc.) or B = Blank.

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewisite Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Taken by	Received Sample	Date
D-79697	D-79697-S-01		2.0 mL	Noel Anthony <i>Anthony Noel</i>	<i>K. J. C. Spauld</i>	07/11/09
					<i>Robert Hunter</i>	07/24/09
				Received for analysis	<i>Mark Miller</i>	07/24/09
	D-79697-L-01		4.0 mL	Noel Anthony <i>Anthony Noel</i>	<i>K. J. C. Spauld</i>	07/11/09
					<i>Robert Hunter</i>	07/24/09
				Received for analysis	<i>Mark Miller</i>	07/24/09

20:00

20:00

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID# M = matrix type (S = sludge L = liquid, G = sorbent tube) NN = sequential # (01, 02, etc.) or B = Blank.

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewisite Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Taken by	Received Sample	Date
D-79699	D 79699-S-01		2.0 mL	Jennings, Donald	K. J. Schuch	07/11/09
					Rachel Hunter	07/24/09
				Received for analysis	John C. Miller	07/24/09
	D-79699-L-01		4.0 mL	Jennings, Donald	K. J. Schuch	07/11/09
					Rachel Hunter	07/24/09
				Received for analysis	John C. Miller	07/24/09

20:00

20:00

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID# M = matrix type (S = sludge, L = liquid, G = sorbent tube), NN = sequential # (01 02 etc.) or B = Blank.

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewisite Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Taken by	Received Sample	Date
D-79700	D-79700-S-01		20 mL	Jennings, Donald	Scott E. Schull	07/11/09
					Robert Hunter	07/24/09
				Received for	Steve Miller	07/24/09 20:00
				analysis		
	D-79700-L-01		40 mL	Jennings, Donald	Scott E. Schull	07/11/09
					Robert Hunter	07/24/09
				Received for	Steve Miller	07/24/09 20:00
				analysis		

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID# M = matrix type (S = sludge, L = liquid, G = sorbent tube) NN = sequential # (01, 02, etc.) or B = Blank.

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewistown Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Taken by	Received Sample	Date
D-79701	D-79701-S-01		2.0 mL	Noel, Anthony <i>Anthony Noel</i>	<i>Kate E. Schuch</i>	07/11/09
					<i>Robert Schuch</i>	07/24/09
				Received for	<i>John C. Bulger</i>	7/24/09
				analysis		
	D-79701-L-01		4.0 mL	Noel, Anthony <i>Anthony Noel</i>	<i>Kate E. Schuch</i>	07/11/09
					<i>Robert Schuch</i>	07/24/09
				Received for	<i>John C. Bulger</i>	07/24/09
				analysis		

20:00

20:00

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID#, M = matrix type (S = sludge L = liquid G = sorbent tube) NN = sequential # (01 02, etc) or B = Blank

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewisite Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Taken by	Received Sample	Date
D-79703	D-79703-S-01		2.0 mL	Noel, Anthony Anthony Noel	Kurt E. Schaefer	07/12/09
					Robert J. Gentry	07/24/09
				Received for analysis	Heidi L. Willey	7/24/09
	D-79703-L-01		4.0 mL	Noel, Anthony Anthony Noel	Kurt E. Schaefer	07/12/09
					Robert J. Gentry	07/24/09
				Received for analysis	Heidi L. Willey	07/24/09

20:00

20:00

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID# M = matrix type (S = sludge L = liquid G = sorbent tube), NN = sequential # (01 02 etc.) or B = Blank

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewisite Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Taken by	Received Sample	Date
D-79705	D-79705-S-01		2.0 mL	Jennings, Donald	Donald Jennings, Jr. & Scott	07/12/09
					Robert Jennings	07/24/09
				Received For	JCA & Williams	07/24/09 20:00
				analysis		
	D-79705-L-01		4.0 mL	Jennings, Donald	Donald Jennings, Jr. & Scott	07/12/09
					Robert Jennings	07/24/09
				Received For	JCA & Williams	07/24/09 20:00
				analysis		

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID#, M = matrix type (S = sludge, L = liquid, G = sorbent tube), NN = sequential # (01, 02, etc.) or B = Blank.

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewisite Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Taken by	Received Sample	Date
D-79711	D-79711-S-01		2.0 mL	Jennings, Donald	Kent E. Shields	07/12/09
					Rachel Hunter	07/24/09
				Received for analysis	Flu G Miller	07/24/09 20:00
	D-79711-L-01		4.0 mL	Jennings, Donald	Kent E. Shields	07/12/09
					Rachel Hunter	07/24/09
				Received for analysis	Flu G Miller	07/24/09 20:00

Sample ID D-XXXXX-M NN where D-XXXXX = Ton Container ID#; M = matrix type (S = sludge, L = liquid G = sorbent tube) NN = sequential # (01, 02, etc.) or B = Blank

Receipt of Sorbent Tubes

The below sorbent tubes were received for analysis on July 24, 2009 at 20 00 hours by Thomas Malloy as part of a secure agent shipment of GA and Lewistite

Sorbent Tube ID	Transparency TC ID
MI-140312	D34998
MI-140591	
MI-140320	D2425
MI-140316	D46304
MI-140315	D36234
MI-140318	D13754
MI-140317	D81037
MI-140599	D43593
MI-140592	
MI-140594	D39003
MI-140595	D45358
MI-140313	D53763
MI-140314	Equipment Blank
MI-140311	Equipment Blank
MI-140593	Equipment Blank


Thomas A Malloy IV

10/2/09
Date

CAR West
DCD One Ton Container Sampling Summary

D Number	Date Sampled	Liquid Pulled	Solid Pulled	Type Container	L/GA Detect	Pressure	Plug Blockage	Remarks
D 2425	7/07/2009	N	N	Transparency	Y (Lewisite)	N	N	Sorbent Tube Pulled #MI-140320
D 13754	7/07/2009	N	N	Transparency	N	N	N	Sorbent Tube Pulled #MI-140318
D 81037	7/08/2009	N	N	Transparency	N	N	N	Sorbent Tube Pulled #MI-140317
D 46304	7/08/2009	N	N	Transparency	Y (Lewisite)	N	N	Sorbent Tube Pulled #MI-140316
* D 34998	7/08/2009	N	N	Transparency	Y (Lewisite)	N	N	Sorbent Tube Pulled #MI-140591
* D 43593	7/08/2009	N	N	Transparency	N	N	N	Sorbent Tube Pulled #MI-140592
D 45358	7/08/2009	N	N	Transparency	Y (Lewisite)	N	N	Sorbent Tube Pulled #595MI-140
D 39003	7/08/2009	N	N	Transparency	N	N	N	Sorbent Tube Pulled #MI-140594
D 36234	7/09/2009	N	N	Transparency	Y (Lewisite)	N	N	Sorbent Tube Pulled #MI-140315
D 53763	7/09/2009	N	N	Transparency	N	N	N	Sorbent Tube Pulled #MI-140313
D 79685	7/09/2009	Y (34 ml)	Y (2 ml)	Lewisite	Y (Lewisite)	N	N	30 ml Sample Pulled 3 Ea 10 ml Bottles
D 49221	7/09/2009	Y (8 ml)	Y (2 ml)	Lewisite	Y (Lewisite)	N	100%	Duplicate Taken

CARA West
DCD One Ton Container Sampling Summary

D Number	Date Sampled	Liquid Pulled	Solid Pulled	Type Container	L/GA Detect	Pressure	Plug Blockage	Remarks
D 79693	7/10/2009	Y (4 ml)	Y (2 ml)	Lewisite	Y (Lewisite)	N	10 %	
D 79701	7/11/2009	Y (4 ml)	Y (2 ml)	Lewisite	Y (Lewisite)	N	90 %	
D 79700	7/11/2009	Y (4 ml)	Y (2 ml)	Lewisite	Y (Lewisite)	N	50 %	
D 79697	7/11/2009	Y (4 ml)	Y (2 ml)	Lewisite	Y (Lewisite)	N	90 %	
D 79699	7/11/2009	Y (4 ml)	Y (2 ml)	Lewisite	Y (Lewisite)	N	50 %	
D 79705	7/12/2009	Y (4 ml)	Y (2 ml)	Lewisite	Y (Lewisite)	N	50 %	
D 79703	7/12/2009	Y (4 ml)	Y (2 ml)	Lewisite	Y (Lewisite)	N	50 %	
D 79711	7/12/2009	Y (4 ml)	Y (2 ml)	Lewisite	Y (Lewisite)	N	25 %	
D 25253	7/15/2009	Y (8 ml)	Y (2 ml)	GA	Y (GA)	N	0 %	Duplicate Taken
D 35248	7/15/2009	Y (4 ml)	Y (2 ml)	GA	Y (GA)	N	0 %	
D 29813	7/16/2009	Y (4 ml)	Y (2 ml)	GA	Y (GA)	N	0 %	

CARA West
DCD One Ton Container Sampling Summary

D Number	Date Sampled	Liquid Pulled	Solid Pulled	Type Container	L/GA Detect	Pressure	Plug Blockage	Remarks
D 51365	7/16/2009	Y (4 ml)	Y (2 ml)	GA	Y (GA)	N	0 %	

* D 34998 Second Sorbent Tube pulled/first tube ran for 6 minutes in lieu of 5 minute requirement New Tube # MI-140312
Original and second tube both shipped to Battelle Labs

* D 43593 Second Sorbent Tube pulled/first tube ran for 6 minutes in lieu of 5 minute requirement New Tube # MI-140599
Original and second tube both shipped to Battelle Labs

Ton Container Sampling Field Log Form

Intrusive Sampling of the Transparency Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	TC Pressure (psi)	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Description (color, consistency)	Heel A Rod Length Inserted	Heel B Angle of Rod Entry	Sample Pumpable (Y/N)	Comments/ Observations
*D-13754	None	D-13754-L-01		0 0 mL	No indication of liquid on sampling tubing upon removal			N	Sampler Noel
	None	D-13754-G-01	20090707 1419-1424	1 0 L @ 200 mL/min					Sorbent Tube MI-140318
*D-2425	None	D-2425-L-01		0 0 mL	No indication of liquid on sampling tubing upon removal			N	Sampler Jennings
	None	D-2425-G-01	20090707 1410-1415	1 0 L @ 200 mL/min					Sorbent Tube MI-140320
*D-46304	None	D-46304-L-01		0 0 mL	No indication of liquid on sampling tubing upon removal			N	Sampler Jennings
	None	D-46304-G-01	20090708 0917-0922	1 0 L @ 200 mL/min					Sorbent Tube MI-140316
*D-81037	None	D-81037-L-01		0 0 mL	No indication of liquid on sampling tubing upon removal			N	Sampler Noel
	None	D-81037-G-01	20090708 0917-0922	1 0 L @ 200 mL/min					Sorbent Tube MI-140317

*These Ton Containers must be the first 5 TC sampled, in the order listed

Sample ID D-XXXXX-M-NN, where D-XXXXX = Ton Container ID# M = matrix type (S = sludge, L = liquid, G = sorbent tube) NN = sequential # (01, 02, etc) or D = Duplicate

Ton Container Sampling Field Log Form

Intrusive Sampling of the Transparency Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	TC Pressure (psi)	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Description (color, consistency)	Heel A Rod Length Inserted	Heel B Angle of Rod Entry	Sample Pumpable (Y/N)	Comments/ Observations
*D-43593	None	D-43593-L-01		0 0 mL	No indication of liquid on sampling tubing upon removal			N	Sampler Jennings
	None	D-43593-G-01	20090708 1117-1123	1 2 L @ 200 mL/min	Sorbent Tube MI-140592				Sorbent Tube MI-140592 Will recollect during borescope
		D-43593-G-01	20090714 0900-0905	1 0 L @ 200 mL/min	Sorbent Tube MI - 140599				
D-34998	None	D-34998-L-01		0 0 mL	Small amount of liquid on sample tubing upon removal			N	Sampler Noel
	None	D-34998-G-01	20090708 1117-1123	1 2 L @ 200 mL/min	Sorbent Tube MI-140591				Sorbent Tube MI-140591 Will recollect Monitoring of interior of TC at 81 STEL Not confirmed with DAAMS tubes
	None	D-34998-G-01	20090710 1520-1525	1 0 L @ 200 mL/min	Sorbent Tube MI-140312				
	None	D-34998-L-D		0 0 mL	Not able to obtain liquid			N	
									DAAMS tubes collected 10 July 09 and confirmed for L
D-39003	None	D-39003-L-01		0 0 mL	No indication of liquid on sampling tubing upon removal			N	Sampler Jennings
	None	D-39003-G-01	20090708 1406-1411	1 0 L @ 200 mL/min					Sorbent Tube MI-140594
D-45358	None	D-45358-L-01		0 0 mL	No indication of liquid on sampling tubing upon removal			N	Sampler Noel
	None	D-45358-G-01	20090708 1408-1413	1 0 L @ 200 mL/min					Sorbent Tube MI-140595

*These Ton Containers must be the first 5 TC sampled, in the order listed

Sample ID D-XXXXX-M-NN, where D-XXXXX = Ton Container ID#; M = matrix type (S = sludge, L = liquid, G = sorbent tube) NN = sequential # (01 02, etc) or D = Duplicate

Ton Container Sampling Field Log Form

Intrusive Sampling of the Transparency Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	TC Pressure (psi)	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Description (color, consistency)	Heel A Rod Length Inserted	Heel B Angle of Rod Entry	Sample Pumpable (Y/N)	Comments/ Observations
D-53763	None	D-53763-L-01		0 0 mL	No indication of liquid on sampling tubing upon removal			N	Sampler Jennings
	None	D-53763-G-01	20090709 0901-0906	1 0 L @ 200 mL/min					Sorbent Tube MI-140313
D-36234	None	D-36234-L-01		0 0 mL	No indication of liquid on sampling tubing upon removal			N	Sampler Noel
	None	D-36234-G-01	20090709 0917-0922	1 0 L @ 200 mL/min					Sorbent Tube MI-140315
									Monitoring of interior of TC at 0.47 STEL
									DAAMS tubes run no confirmation indicated for lewisite

*These Ton Containers must be the first 5 TC sampled, in the order listed

Sample ID D-XXXXX-M-NN, where D-XXXXX = Ton Container ID#, M = matrix type (S = sludge L = liquid G = sorbent tube), NN = sequential # (01 02, etc) or D = Duplicate

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewisite Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	TC Pressure (psi)	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Description (color, consistency)	Heel A Rod Length Inserted	Heel B Angle of Rod Entry	Sample Pumpable (Y/N)	Comments/ Observations
D-49221	None	D-49221-S-01	20090709 1335	2 0 mL	Black, Tarry	7			Sampler Noel - Plug well opening 100% blocked
	None	D-49221-L-01	20090709 1347	4 0 mL	Black, Water Like			Y	
	None	D-49221-L-D	20090709 1414	4 0 mL	Black, Water Like			Y	
D-79685	None	D-79685-S-01	20090709 1423	2 0 mL	Black, Tar Like	8			Sampler Jennings
	None	D-79685-L-01	20090709 1336	4 0 mL	Black, Water Like			Y	
	None	D-79685-L-02 See Comments	20090709 1353	30 0 mL	Black, Water Like			Y	D-79685-L-02, D-79685-L-03, D-79685-04 each 10 mL volume
D-79693	None	D-79693-S-01	20090710 1104	2 0 mL	Black, Tar Like	4			Sampler Jennings - Plug well opening 10% blocked
	None	D-79693-L-01	20090710 1114	4 0 mL	Black, Water Like			Y	
D-79697	None	D-79697-S-01	20090711 1450	2 0 mL	Black, Tar like	3			Sampler Noel - Plug well opening 90% blocked
	None	D-79697-L-01	20090711 1510	4 0 mL	Black, Water Like			Y	

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID#, M = matrix type (S = sludge, L = liquid G = sorbent tube) NN = sequential # (01, 02 etc) or D = Duplicate

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewistite Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	TC Pressure (psi)	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Description (color, consistency)	Heel A Rod Length Inserted	Heel B Angle of Rod Entry	Sample Pumpable (Y/N)	Comments/ Observations
D-79699	None	D-79699-S-01	20090711 1447	2 0 mL	Black, Tar like	5			Sampler Jennings - Plug well opening 50% blocked
	None	D-79699-L-01	20090711 1459	4 0 mL	Black, Water Like			Y	
D-79700	None	D-79700-S-01	20090711 1010	2 0 mL	Black, Tar like	4			Sampler Jennings - Plug well opening 50% blocked
	None	D-79700-L-01	20090711 1032	4 0 mL	Black, Water Like			Y	
D-79701	None	D-79701-S-01	20090711 1011	2 0 mL	Black, Tar like	4			Sampler Noel - Plug well opening 90% blocked
	None	D-79701-L-01	20090711 1025	4 0 mL	Black, Water Like			Y	
D-79703	None	D-79703-S-01	20090712 1000	2 0 mL	Black, Tar Like	3			Sampler Noel - Plug well opening 50% blocked
	None	D-79703-L-01	20090712 1008	4 0 mL	Black, Water Like			Y	

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID# M = matrix type (S = sludge L = liquid G = sorbent tube) NN = sequential # (01 02, etc.) or D = Duplicate

Ton Container Sampling Field Log Form

Intrusive Sampling of the Lewisite Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	TC Pressure (psi)	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 mL	Sample Description (color, consistency)	Heel A Rod Length Inserted	Heel B Angle of Rod Entry	Sample Pumpable (Y/N)	Comments/ Observations
D-79705	None	D-79705-S-01	20090712 0952	2.0 mL	Black, Tar Like	4			Sampler Jennings - Plug well opening 50% blocked
	None	D-79705-L-01	20090712 1001	4.0 mL	Black, Water Like			Y	
D-79711	None	D-79711-S-01	20090712 1423	2.0 mL	Black, Tar Like	4			Sampler Jennings - Plug well opening 25% blocked
	None	D-79711-L-01	20090712 1428	4.0 mL	Black, Water Like			Y	

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID#, M = matrix type (S = sludge, L = liquid, G = sorbent tube), NN = sequential # (01, 02 etc.) or D = Duplicate

Ton Container Sampling Field Log Form
Intrusive Sampling of the GA Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	TC Pressure (psi)	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 ml	Sample Description (color, consistency)	Heel A Rod Length Inserted	Heel B Angle of Rod Entry	Sample Pumpable (Y/N)	Comments/ Observations
D-25253	None	D-25253-S-01		2 0 mL	Unable to collect solid	9			Sampler - Jennings
	None	D-25253-L-01	20090715 1350	4 0 mL	Dark Brown, Light Oil			Y	Very little residue on solid sample probe upon removal
	None	D-25253-L-D	20090715 1404	4 0 mL	Dark Brown, Light Oil			Y	
D-35248	None	D-35248-S-01		2 0 mL	Unable to collect solid	8			Sampler - Noel
	None	D-35248-L-01	20090715 1352	4 0 mL	Dark Brown, Motor Oil			Y	
D-29813	None	D-29813-S-01	20090716 1019	~ 1 0 mL	Dark Brown, Wet-Sandy	5			Sampler - Jennings Received information that Battelle Labs needed more sample than was collected. Mr Hubanks spoke with Inventory and CARA will destroy sample and certificate of destruction will be completed. Inventory Shields and CARA Hatcher will witness destruction of TC D-29813-S-01
	None	D-29813-L-01	20090716 1029	4 0 mL	Dark Brown, Motor Oil			Y	

Sample ID D-XXXXX-M-NN, where D-XXXXX = Ton Container ID#, M = matrix type (S = sludge, L = liquid, G = sorbent tube), NN = sequential # (01, 02, etc.) or D = Duplicate

Ton Container Sampling Field Log Form
Intrusive Sampling of the GA Ton Container Inventory for TOCDF

Ton Container Number Example D-XXXXX	TC Pressure (psi)	Sample ID Number Example D-XXXXX-M-NN	Collection Ordinal Date/Time Example 2009001-0800	Sample Volume Example 2 ml	Sample Description (color, consistency)	Heel A Rod Length Inserted	Heel B Angle of Rod Entry	Sample Pumpable (Y/N)	Comments/ Observations
D-51365	None	D-51365-S-01		2.0 mL	Unable to collect solid	7			Sampler - Noel
	None	D-51365-L-01	20090716 1036	4.0 mL	Dark Brown, Motor Oil			Y	

Sample ID D-XXXXX-M-NN where D-XXXXX = Ton Container ID# M = matrix type (S = sludge, L = liquid, G = sorbent tube) NN = sequential # (01, 02, etc.) or D = Duplicate

MRC JJ-212-PB

MRC
4D Box

70.6 Lbs.

76.0 Lbs.

146.6

D-79685-L-01 4 mL

D-79685-L-02 10 mL

D-79685-L-03 10 mL

D-79685-L-04 10 mL

D-79699-L-01 4 mL

D-79700-L-01 4 mL

D-79701-L-01 4 mL

D-79703-L-01 4 mL

D-79705-L-01 4 mL

D-79693-L-01 4 mL

D-79711-L-01 4 mL

D-79697-L-01 4 mL

Total 66 mL

D-79699-S-01 2 mL

D-79700-S-01 2 mL

D-79701-S-01 2 mL

D-79703-S-01 2 mL

D-79705-S-01 2 mL

D-79685-S-01 2 mL

Total 12 mL

Container Total 78 mL combined liquid/solid

MRC JJ-079-PB

MRC 65.6 Lbs.
4D Box 76
141.6

D-49221-S-01 2 mL
D-79693-S-01 2 mL
D-79697-S-01 2 mL
D-79711-S-01 2 mL Total 8 mL

D-49221-L-01 4 mL
D-49221-L-D 4 mL Total 8 mL

Container Total 16 mL combined liquid/solid

Sorbent Tube

D-34998-G-01 MI-140591 1.2L@200ml/min

D-34998-G-01 MI-140312 1.0L@200ml/min

Added upon repack of MRC JJ-040-PB into
JJ-079-PB

D-2425-G-01 MI-140320 1.0L@200ml/min

D-46304-G-01 MI-140316 1.0L@200ml/min

D-36234-G-01 MI-140315 1.0L@200ml/min

LSC 94C019

LSC 44.6 Lbs.
4D Box 63.2 Lbs.
107.8

D-25253-L-01 4 mL

D-25253-L-D 4 mL

D-35248-L-01 4 mL

D-29813-L-01 4 mL

D-51365-L-01 4 mL

Container Total 20 mL

BATTELLE HMRC CA STOCK RECORD CARD NO W001-1-1

Page 1

CA GA

LOT No D-25253-L-01

UNIT OF ISSUE g

VIAL TARE WT 25.5486

Tare weight added after first use if necessary or enter NA

ROOM No VAULT

HOOD 12

Vault ☐ Refrigerator ☒ W1

VIAL(s) ID 1

DD1911 No W90BDL - 09205 - 3002/3003

DATE	TD#	PROJECT #	LID TARE	STARTING	FINAL	GAINS	LOSS	BALANCE	SIGNATURE
2/18/09	-	X from W001	3.1188	28.1674	32.9594	4.2920	-	4.2920	Bruce H. Tully
2/28/09	3017	C117	3.1188	32.9594	32.8114	-	0.1480	4.1440	Bruce H. Tully
2/28/09	3017	6006328-02	3.1188	32.8114	32.6002	-	0.2112	3.9328	Bruce H. Tully
7/21/09	INV			32.6002				3.9328	Bruce H. Tully
8/12/09	3019	6006328-02	3.1188	32.6002	32.5874	-	0.0128	3.9200	Bruce H. Tully
8/27/09	INV			32.5874				3.9200	Bruce H. Tully
9/11/09	3027	6006328-03	3.1188	32.5874	32.5748	-	0.0126	3.9074	A. Stacy
9/11/09	3028	6006328-03	3.1188	32.5748	32.3984	-	0.1764	3.7310	A. Stacy
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COMMENTS

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BATTELLE HMRC CA STOCK RECORD CARD NO W002-1-1 Page 1

CA GA

LOT No D-25253-L-D

UNIT OF ISSUE g

VIAL TARE WT 05 6539

Tare weight added after first use if necessary or enter NA

ROOM No VAULT

HOOD 12

Vault ☐
Refrigerator ☒ W1

VIAL(s) ID 1

DD1911 No W90 BDL-09205-3002/3003

DATE	TD#	PROJECT #	LID TARE	STARTING	FINAL	GAINS	LOSS	BALANCE	SIGNATURE
7/28/09	-	<i>1/2 from W002</i>	3.1482	28.8021	33.0941	4.2920	-	4.2920	<i>Ben H. Tully</i>
7/28/09	3017	<i>C117</i>	3.1482	33.0941	32.2852	-	0.8089	3.4831	<i>Ben H. Tully</i>
7/28/09	3017	<i>6006328-02</i>	3.1482	32.2852	32.0977	-	0.1875	3.2956	<i>Ben H. Tully</i>
7/29/09	INV			32.0977				3.2956	<i>Ben H. Tully</i>
8/12/09	3019	<i>6006328-02</i>	3.1482	32.0977	32.0831	-	0.0146	3.2810	<i>A. Stacy</i>
8/21/09	INV			32.0831				3.2810	<i>A. Stacy</i>
9/11/09	3027	<i>6006328-03</i>	3.1482	32.0831	32.0602	-	0.0229	3.2581	<i>A. Stacy</i>
9/11/09	3028	<i>6006328-03</i>	3.1482	32.0602	31.8868	-	0.1734	3.0847	<i>A. Stacy</i>
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COMMENTS

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BATTELLE HMRC CA STOCK RECORD CARD NO

Page

CA 6A LOT No D-35248-L-01 UNIT OF ISSUE g VIAL TARE WT 25.5169
Tare weight added after first use if necessary or enter NA

ROOM No Vault HOOD 12 Vault ☐ Refrigerator ☒ VIAL(s) ID 1

DD1911 No W90BDL-09205-3002/3003

[illegible]

COMMENTS

All blanks must contain an entry

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SRC originated by

BATTELLE HMRC CA STOCK RECORD CARD NO

W004-1-1

Page 1

CA GA

LOT No D-29813-L-01

UNIT OF ISSUE g

VIAL TARE WT 25.4095

Tare weight added after first use if necessary or enter NA

ROOM No VAULT HOOD 12 Vault ☐ Refrigerator ☒ w/ VIAL(s) ID 1

DD1911 No W90BDL-09205-3002/3003

DATE	TD#	PROJECT #	LID TARE	STARTING	FINAL	GAINS	LOSS	BALANCE	SIGNATURE
7/28/09	-	X from W004	3.0861	28.4956	32.7876	4.2920	-	4.2920	Brian K. Foley
7/28/09	3017	C117	3.0861	32.7876	33.0714	0.2838	-	4.5758	Brian K. Foley
7/28/09	3017	G006328-02	3.0861	33.0714	32.8803	-	0.1911	4.3847	Brian K. Foley
7/29/09	INV			32.8803				4.3847	U. Stacey
8/12/09	3019	G006328-02	3.0861	32.8803	32.8402	-	0.0401	4.3446	U. Stacey
8/27/09	INV			32.8402				4.3446	U. Stacey
9/1/09	3027	G006328-03	3.0861	32.8402	32.7729	-	0.0673	4.2773	U. Stacey
9/1/09	3028	G006328-03	3.0861	32.7729	32.6366	-	0.1363	4.1409	U. Stacey
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COMMENTS

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BATTELLE HMRC CA STOCK RECORD CARD NO. W005-1-1 Page 1

CA GA LOT No D-51365-L-01 UNIT OF ISSUE g VIAL TARE WT 25 3227
Tare weight added after first use if necessary or enter NA

ROOM No VAULT HOOD 12 Vault ☐ Refrigerator ☒ W1 VIAL(s) ID 1

DD1911 No W90BDL-09205-3002/3003

DATE	TD#	PROJECT #	LID TARE	STARTING	FINAL	GAINS	LOSS	BALANCE	SIGNATURE
7/28/09	-	Xfer from W005	3.1944	28.5171	32.8091	4.2920	-	4.2920	Bruce R. Taylor
7/28/09	3017	C117	3.1944	32.8091	32.5355	-	0.2736	4.0184	Bruce R. Taylor
7/28/09	3017	6006328-02	3.1944	32.5355	32.3322	-	0.2033	3.8151	Bruce R. Taylor
7/29/09	INV			32.3322				3.8151	E. R. Taylor
8/12/09	3019	6006328-02	3.1944	32.3322	32.3171	-	0.0151	3.8000	A. Stacey
8/12/09	INV			32.3171				3.8000	A. Stacey
9/11/09	3027	6006328-03	3.1944	32.3171	32.2960	-	0.0211	3.7789	A. Stacey
9/11/09	3028	6006328-03	3.1944	32.2960	32.1694	-	0.1266	3.6523	A. Stacey
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COMMENTS

All blanks must contain an entry

Wool

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L

D-49221-S-01

UNIT OF ISSUE

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

Vault

12

Vault Refrigerator

B

✓ w2

VIAL(s) ID

1

DD1911 No W90BDL - 09205 - 3001

COMMENTS All agent switched from ml to g - see 7/27/09

All blanks must contain an entry

Controlled Document HMRC 0077 Feb 2007

SRC originated by

But

BATTELLE HMRC CA STOCK RECORD CARD NO

W007

Page 1

CA

L

LOT No

D-49221-L-01

UNIT OF ISSUE

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

ROOM No

VAULT

HOOD 12

Vault ☐ Refrigerator ☒

W2

VIAL(s) ID

1

DD1911 No

W90BDL-09205-3001

DATE	TD#	PROJECT #	LID TARE	STARTING	FINAL	GAINS	LOSS	BALANCE	SIGNATURE
7/24/09	RCVD	1 x 4	—	—	4	4	—	4	Bruce Capwell
7/28/09	—	C117	—	—	7.5200	7.5200	—	7.5200	Bruce Capwell
7/28/09	3018	6006328-02	—	26.7525	26.5316	—	0.2209	7.2991	Bruce Capwell
7/29/09	INV	—	—	26.5316	—	—	—	7.2991	Bruce Capwell
8/12/09	3019	6006328-02	—	26.5316	26.5110	—	0.0206	7.2785	Bruce Capwell
8/27/09	INV	—	—	26.5110	—	—	—	7.2785	Bruce Capwell
9/12/09	3029	6006328-03	—	26.5110	26.2737	—	0.2373	6.9812	A. Stacey
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COMMENTS

All agent switched from mL to g - Mr 7/28/09

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BATTELLE HMRC CA STOCK RECORD CARD NO

W008

Page 1

CA

L

LOT No

D-49221-L-D

UNIT OF ISSUE

W 7/28/09

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

ROOM No

VAULT

HOOD

12

Vault Refrigerator

☐ W2

VIAL(s) ID

1

DD1911 No

W90BDL-09205-3001

DATE	TD#	PROJECT #	LID TARE	STARTING	FINAL	GAINS	LOSS	BALANCE	SIGNATURE
7/24/09	RCVD	1 x 4	—	—	4	4	—	4	Bruce Capelle
7/28/09	—	C117	—	—	2.5200	2.5200	—	2.5200	Bruce Capelle
7/28/09	30/8	6006328-02	—	28.6071	28.4071	—	0.2000	2.3200	Bruce Capelle
7/29/09	INV	—	—	28.4071	—	—	—	7.3200	Bruce Capelle
8/12/09	30/9	6006328-02	—	28.4071	28.3709	—	0.0362	7.2832	Bruce Capelle
8/17/09	INV	—	—	28.3709	—	—	—	7.2832	Bruce Capelle
9/2/09	30/9	6006328-03	—	28.3709	27.9970	—	0.3739	6.5354	Bruce Capelle
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COMMENTS

All agent switched from mL to g - Bu 7/28/09

All blanks must contain an entry

BATTELLE HMRC CA STOCK RECORD CARD NO

W009

Page 1

CA

L

LOT No

D-79685-S-01

UNIT OF ISSUE

ML 7/27/09 g

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

ROOM No

VAULT

HOOD 12

Vault ☐
Refrigerator ☒

W2

VIAL(s) ID

1

DD1911 No

W90BDL-09205-3001

DATE	TD#	PROJECT #	LID TARE	STARTING	FINAL	GAINS	LOSS	BALANCE	SIGNATURE
7/24/09	REVD	1 X 2	—	—	2	2	—	2	Bruce Capwell
7/27/09	—	C117	—	—	3.7600	3.7600	—	3.7600	Bruce Capwell
7/27/09	30/5	6006328-02	—	23.4790	22.8643	—	0.6147	3.1453	Bruce Capwell
7/29/09	INV	—	—	72.8643	—	—	—	3.1453	Bruce Capwell
8/27/09	INV	—	—	72.8643	—	—	—	3.1453	Bruce Capwell
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COMMENTS All agent amount switched from ML to g. - 7/27/09 ML

All blanks must contain an entry

Bruce

Wolo

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L

D-79685-L-01

me 2/29/09

NA

NA

Vault

Vault

✓ W2

1

DD1911 No

[illegible]

COMMENTS

All blanks must contain an entry

ROOM No VAULT HOOD 12 Vault Refrigerator ☒ 43 VIAL(s) ID 1

[illegible]

SRC originated by *BW*

W013

Page

CA

LOT No D-79693-L-01

UNIT OF ISSUE

9 VIAL TARE WT NA

Tare weight added after first use if necessary or enter NA

ROOM No

Vault

HOOD 12

Vault ☐
Refrigerator ☒ **W2** **VIAL(s) ID**

DD1911 No W90BDL- 09205- 3001

COMMENTS All agent switched from wk to g - Rn 7/28/09

All blanks must contain an entry

Page

LOT No

D-79697-S-01

UNIT OF ISSU

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

ROOM No

Vault

HOOD 12

Vault ☐
Refrigerator ☒

W3

VIAL(s) ID

1

DD1911 No W90BDL - 09205 - 3001

COMMENTS All agent amount switched from ml to g - Mr 7/27/09

All blanks must contain an entry

Buy

W015

Page

CA

L

LOT No

D-79697-L-01

UNIT OF ISSUE

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

ROOM No

VAULT

HOOD

12

Vault ☐

Refrigerator ☒

✓ W3 VIAL(s) ID

1

DD1911 No

W90BDL - 09205 - 3001

[illegible]

COMMENTS All agent switched from ml to g - Bcr 7/26/09

All blanks must contain an entry

Page 1

UNIT OF ISSUE *pk 9* VIAL TARE WT *NA*

ROOM No VAULT HOOD 12 Vault ☐ Refrinator ☒ w3 VIAL(s) ID 1

[illegible]

COMMENTS	All asset amount switched from unit to g - see 7/27/09 7/24/09 in 7/27/09 All b	
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All blanks must contain an entry

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NA

Tare weight added after first use if necessary or enter NA

✓ W3 VIAL(s) ID

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1/20/81

All agent switched from WL to g - Bur 7/28/04

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Buy

Tare weight added after first use if necessary or enter NA

1

Buk

W019

1

L

D-79700-L-01

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

Vault

12

Vault ☐

Refrigerator ☒

W3

VIAL(s) ID

1

W90BDL - 09205 - 3001

Bruce Capwell
 A-Stack
 A-Stack
 Eng. Phil
 A-Stack
 (Bunn)
 A-Stack

All agent switched from ml to g - Rec 7/28/09

All blanks must contain an entry

BATTELLE HMRC CA STOCK RECORD CARD NO

W020

Page 1

CA L

LOT No D-79701-S-01

UNIT OF ISSUE ^{W 7/27/09} 9

VIAL TARE WT NA

Tare weight added after first use if necessary or enter NA

ROOM No VAULT HOOD 12 Vault ☐ Refrigerator ☒ W4 VIAL(s) ID 1

DD1911 No W90BDL - 09205-3001

DATE	TD#	PROJECT #	LID TARE	STARTING	FINAL	GAINS	LOSS	BALANCE	SIGNATURE
7/24/09	RCVD	1 x 2	—	—	2	2	—	2	Bruce Campbell
7/27/09	—	C117	—	37600	37600	—	—	37600	A. Stacey
7/27/09	3015	6006328-02	—	714076	714076	—	0.1742	3.5858	A. Stacey
7/29/09	INV	—	—	71.4076	—	—	—	3.5858	Bruce Campbell
8/27/09	INV	—	—	71.4076	—	—	—	3.5858	Bruce Campbell
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COMMENTS All agent amounts switched from mL to g - BUC 7/27/09

All blanks must contain an entry

BATTELLE HMRC CA STOCK RECORD CARD NO.

W021

Page 1

CA

L

LOT No D-79701-L-01

UNIT OF ISSUE

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

ROOM No

VAULT

HOOD

12

Vault ☐
Refrigerator ☒

W4

VIAL(s) ID

1

DD1911 No

W90BDL-09205-3001

DATE	TD#	PROJECT #	LID TARE	STARTING	FINAL	GAINS	LOSS	BALANCE	SIGNATURE
7/24/09	RCVD	1 x 4	—	—	4	4	—	4	Bum Campbell
7/28/09	—	CH7	—	—	75200	75200	—	75200	A. Stacy
7/28/09	3018	6006328-02	—	2682016	266258	—	0.1948	7.3252	A. Stacy
7/29/09	INV	—	—	266258	—	—	—	7.3252	A. Stacy
8/12/09	3020	6006328-02	—	266258	265890	—	0.0368	7.2884	A. Stacy
8/27/09	INV	—	—	265890	—	—	—	7.2884	A. Stacy
9/21/09	3029	6006328-03	—	265890	263739	—	0.2151	7.0733	A. Stacy
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COMMENTS

All blanks must contain an entry

Buc

W022

Page

CA

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LOT No

D-79703-S-01

UNIT OF ISSUE

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

ROOM No.

VAULT

HOOD

12

Vault Refrigerator

☒ W4

VIAL(s) ID

1

DD1911 No

W90BDL - 09205 - 3001

[illegible]

COMMENTS

All agent switched from ml to g - Mx 7/27/09

All blanks must contain an entry

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Bye

BATTELLE HMRC CA STOCK RECORD CARD NO

W023

Page 1

CA

L

LOT No

D-79703-L-01

UNIT OF ISSUE

W 1/26/09

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

ROOM No

VAULT

HOOD

12

Vault ☐
Refrigerator ☒

W4

VIAL(s) ID

1

DD1911 No

W90BDL-09205-3001

DATE	TD#	PROJECT #	LID TARE	STARTING	FINAL	GAINS	LOSS	BALANCE	SIGNATURE
7/24/09	RLVD	1 x 4	—	—	4	4	—	4	Bruce Capwell
7/28/09	—	C117	—	—	75200	75200	—	75200	A. Steacy
7/28/09	3018	6006328-02	—	28.3501	28.1627	—	0.1937	7.3263	A. Steacy
7/29/09	INV	—	—	28.1627	—	—	—	7.3263	A. Steacy
8/12/09	3020	6006328-02	—	28.1627	28.1378	—	0.0249	7.3014	A. Steacy
8/27/09	INV	—	—	28.1378	—	—	—	7.3014	A. Steacy
9/12/09	3029	6006328-03	—	28.1378	27.9650	—	0.0228	7.0886	A. Steacy
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COMMENTS

All agent switched from mL to g - Buz 7/26/09

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BATTELLE HMRC CA STOCK RECORD CARD NO

W024

Page 1

CA

L

LOT No

D-79705-S-01

UNIT OF ISSUE

ml 7/27/09

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

ROOM No

VAULT

HOOD

12

Vault ☐ Refrigerator ☒

WS

VIAL(s) ID

1

DD1911 No

W90BDL-09205-3001

DATE	TD#	PROJECT #	LID TARE	STARTING	FINAL	GAINS	LOSS	BALANCE	SIGNATURE
7/24/09	RCVD	1 x 2	—	—	2	2	—	2	Bruce Campbell
7/27/09	—	C117	—	—	3.7600	3.7600	—	3.7600	A. Stagen
7/27/09	3015	600632802	—	73.533	73.3238	—	0.2095	3.5505	A. Stagen
7/29/09	INV	—	—	73.3238	—	—	—	3.5505	—
8/27/09	INV	—	—	73.3238	—	—	—	3.5505	—
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COMMENTS

Act agent switched from ml to g - ml 7/27/09

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LOT No

D-79705-L-01

UNIT OF ISSUE ~~XXXX~~

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

ROOM No

VAULT

HOOD

12

Vault ☐
Refrigerator ☒

☒ W5

VIAL(s) ID

1

DD1911 No

W90BDL-09205-3001

COMMENTS

All agent switched from ul to g - Bu 7/28/09

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Buy

1

Tare weight added after first use if necessary or enter NA

1

Boat

BATTELLE HMRC CA STOCK RECORD CARD NO

W027

Page 1

CA

L

LOT No D-79711-L-01

UNIT OF ISSUE ^{ml 7/28/09} ~~ML~~ g

VIAL TARE WT

NA

Tare weight added after first use if necessary or enter NA

ROOM No VAULT HOOD 12 Vault ☐ Refrigerator ☒ W5 VIAL(s) ID 1

DD1911 No W90BDL-09205-3001

DATE	TD#	PROJECT #	LID TARE	STARTING	FINAL	GAINS	LOSS	BALANCE	SIGNATURE
7/24/09	RCLD	1 x 4	—	—	4	4	—	4	Bruce Campbell
7/28/09	—	C117	—	7.5200	7.5200	—	—	7.5200	A. Stacy
7/28/09	3018	6006328-02	—	25.8515	25.6520	—	0.1995	7.3205	A. Stacy
7/29/09	INV	—	—	25.6520	25.6520	—	—	7.3205	—
8/12/09	3020	6006328-02	—	25.6520	25.6810	—	0.0210	7.2995	A. Stacy
8/27/09	INV	—	—	25.6310	—	—	—	7.2995	—
9/2/09	3029	6006328-03	—	25.6310	25.4197	—	0.2113	7.0882	A. Stacy
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COMMENTS All agent switched from ml to g - Bru 7/28/09

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Appendix B

Sampling Plan

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REPLY TO
ATTENTION OF

DEPARTMENT OF THE ARMY
US ARMY CHEMICAL MATERIALS AGENCY
TOOELE CHEMICAL AGENT DISPOSAL FACILITY
11620 STARK ROAD
STOCKTON, UTAH 84071

HAND DELIVERED

JUN 11 2009

**UTAH DIVISION OF
SOLID & HAZARDOUS WASTE**

June 4, 2009

Tooele Chemical Agent Disposal Facility

PM0272-09

SUBJECT GA/Lewisite (GA/L) Disposal Facility, Ton Container Sampling Program Plan,
June 1, 2009, Revision 1, EPA ID UT 5210090002

Mr Dennis Downs, Director
Utah Department of Environmental Quality
Division of Solid and Hazardous Waste
PO Box 144880
288 North 1460 West
Salt Lake City, Utah 84114-4880

Dear Mr Downs

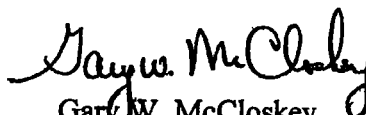
Enclosed, for your information and to support future permitting efforts at TOCDF, is the final GA/L Disposal Facility Ton Container Sampling Plan, Revision 1, dated June 1, 2009


Revision 1 contains the TOCDF responses to the DSHW comments as discussed during a phone conference with members of the DSHW staff. A summary of the comment responses is also included. TOCDF believes that this Revision incorporates the DSHW comments and that these comments have been resolved to DSHW's satisfaction.

The current target date for this sampling effort is July 7, 2009. If you have additional comments or questions, please contact us as soon as possible.

If you have any questions regarding this issue, please contact Ms Sheila R. Vance at (435) 833-7577 or Mr Trace Salmon at (435) 833-7428.

Sincerely,


Gary W. McCloskey
EG&G Defense Materials, Inc
*CERTIFICATION STATEMENT


Thaddeus A. Ryba, Jr
TOCDF Site Project Manager
*CERTIFICATION STATEMENT

Enclosure

* I CERTIFY UNDER PENALTY OF LAW THAT THIS DOCUMENT AND ALL ATTACHMENTS WERE PREPARED UNDER MY DIRECTION OR SUPERVISION IN ACCORDANCE WITH A SYSTEM DESIGNED TO ASSURE THAT QUALIFIED PERSONNEL PROPERLY GATHER AND EVALUATE THE INFORMATION SUBMITTED BASED ON MY INQUIRY OF THE PERSON OR PERSONS WHO MANAGE THE SYSTEM OR THOSE PERSONS DIRECTLY RESPONSIBLE FOR GATHERING THE INFORMATION. THE INFORMATION SUBMITTED IS TO THE BEST OF MY KNOWLEDGE AND BELIEF TRUE, ACCURATE AND COMPLETE. I AM AWARE THAT THERE ARE SIGNIFICANT PENALTIES FOR SUBMITTING FALSE INFORMATION INCLUDING THE POSSIBILITY OF FINE AND IMPRISONMENT FOR KNOWING VIOLATIONS.

DSHW Draft Comments on GA/Lewisite/Transparency TC Sampling Plan
Summary of Comment Resolution Discussed via Telephone
March 26, 2009, updated responses June 1, 2009

- 1 Why are no solids samples going to be collected from the GA ton containers? Unless all of the GA ton containers can be documented to be free of solids, solid samples should be collected
Based on historical data and characterization efforts to date, TOCDF has no reason to suspect the presence of solids in the GA

Resolution DSHW would be comfortable if we could determine a mechanism to evaluate the TCs for the presence of solids and obtain a sample if we do encounter solids

The plan has been amended as follows to allow for the collection of solids as in Section 1.2 as follows

- “Objective 1 To collect liquid samples at the 25%, 50%, and 75% fill levels (horizontal TC orientation) and composite these samples into a single liquid sample for each of the GA TCs. If present, a sludge sample will be collected from each of the GA TCs with effort taken to ensure that only sludge (no liquid) is collected
- The GA will be analyzed for agent purity, density, pH, Health Risk Assessment (HRA) metals, chlorobenzene content, and tentatively identified compounds (TICs) as identified by GC/MS
 - Each sludge sample, if present, will be analyzed for HRA metals
- Objective 2 To collect liquid samples at the 25%, 50%, and 75% fill levels (horizontal TC orientation) and composite these samples into a single liquid sample for each of the Lewisite TCs. If present, a sludge sample will be collected from each of the Lewisite TCs with effort taken to ensure that only sludge (no liquid) is collected
- Each liquid Lewisite sample will be analyzed for agent purity (L1, L2, L3), density, pH, HRA metals, and tentatively identified compounds (TICs) as identified by GC/MS
 - Each sludge sample, if present, will be analyzed for HRA metals”
- 2 Objectives one & two For later waste characterization sampling for DSHW, a determination if one liquid sample is representative of the entire ton container must be determined for both GA and Lewisite. Is additional data such as PINS being performed? Composite sampling should not be performed in this sampling phase if a determination is supposed to be made that all liquid samples are the same no matter where they are collected
- DSHW explained their preference for obtaining samples/analysis of each phase as opposed to compositing the sample, due to a desire to have the same information that was available on HD TCs to demonstrate “one liquid sample” was sufficient to characterize the entire TC. It was explained to DSHW that the data quality objective for this sampling effort is not to make such a demonstration, since the information required to support treatment objectives is very different. In the case of the HD TCs, we knew we would not have sufficient Hg DRE with our current PAS and due to the significant variation of Hg concentrations in the HD TC population, we needed a mechanism to screen every TC. The sampling approach designed for that particular situation was done with the acknowledgement that the Hg concentration in the heels of the TCs was non-homogeneous. Therefore we had a specific need to demonstrate that one liquid sample was adequate to characterize each TC as either “low Hg” or “high Hg”, such that we could determine the appropriate treatment approach (either before or after Hg abatement was installed on the PAS). Conversely, in the case of the GA/L sampling, we do not expect any metals of concern in the GA and we know we will have to design a PAS that will provide sufficient Hg and As abatement for the Lewisite. Compositing each sample provides for a more representative analysis of what will*

actually be fed to the incinerator, and therefore is more appropriate for our data quality objective with respect to needing characterization information to support waste treatment objectives
Resolution DSHW accepted composite sampling for the reasons stated above

- 3 Are other incineration parameters analyzed such as BTU?
TOCDF personnel explained the basis for not analyzing incineration parameters such as BTU, in the case of BTU – we know the facility design will assure necessary combustion regardless of potential variation in the BTU value of the waste feed
Resolution DSHW accepted our response

- 4 All phases of the ton containers contents, both GA and Lewisite should be analyzed for volatiles, SVOC, metals, pH and density
See above comment response #2 regarding “all phases” requiring analysis With respect to the analysis to be performed, quantitative SVOC and VOC analysis are not planned, a mass spectral library search will identify “the Tentatively Identified Compounds” (TICs) and this will provide sufficient characterization information to support treatment Prior to processing in the LIC, a surrogate test will be performed using a compound that, based on EPA’s incinerability index, is more difficult to burn than Lewisite (likely chlorobenzene) Therefore, there is no need for a quantitative analysis of SVOCs or VOCs
Resolution DSHW would like us to include a more thorough description in the Plan of the analytical/TIC identification process

This comment has been addressed in section 3 2 2 under subheading “Agent purity and TICs for Lewisite liquid samples” as follows

“Analysis will be performed by GC/MS per SOP HMRC IV-055 to determine the retention time (RT) of Lewisite 1 and 2, identified as their thioether derivatives, and Lewisite 3 A NIST mass spectral library will be used to evaluate samples for TICs Reports will be generated using ChemStation software “

- 5 MINICAMS is not an approved quantitation method for Lewisite, suggest using DAAMS tubes
TOCDF personnel clarified the purpose of the MINICAMS as a qualitative screening tool for the Transparency TCs, as well as the additional follow-on quantitative analysis, as appropriate
Resolution DSHW accepted TOCDF’s response and acknowledged that the purpose of this approach was more specific to Treaty requirements than to waste characterization

- 6 Provide a description of the actual sampling procedure, type of sampling device, mechanism for determination if liquid is present etc
We need to develop more details in coordination with CARA West – DSHW has no specific concerns, but wants to make sure they understand how the samples will be collected, to assure representativeness, etc
Resolution TOCDF committed to developing more detail, sharing such detail with DSHW, and incorporating it into the Plan, when available

The following text has been added to section 3 1 3 to address this concern Also, the CARA West SOP’s to cover sampling procedures are included in Appendix A

“EG&G will notify DSHW at least 72 hours in advance of the initiation of Area 10 GA/Lewisite sampling operations DCD will notify OPCW as required Sample collection, packaging and shipping to the HMRC will be accomplished by CARA West IAW Army Regulations

CARA West will arrive with their specialized equipment consisting of glove boxes, air filters, generators, decontamination facilities, agent monitoring, and other supplies necessary to collect the agent samples to complete the sampling program. Air monitoring stations will be established using instruments operating under existing CARA WEST Precision and Accuracy studies. CARA West will perform alternate baseline monitoring studies (Per CMA LMQAP Section 10.4) while on-site to and submit them to CMA for approval.

After confirmation that the glove boxes are working properly, the GA, GA/UCON, Lewisite, and "transparency" TCs will be received from Area 10 and placed in the glovebox. The TCs will be positioned such that the heel weather mark is toward the bottom (horizontal TC orientation), the storage arrow marked by Area 10 is visible at the top, and the "working" plug is at the top. Preliminary physical examination of the TCs (5/19/09) suggests the presence/absence of heel material for the TCs. This information will ensure that the sampling effort is conducted efficiently.

Prior to placement in the glovebox, the CARA West Sampling Operators will measure and record the external temperature of each TC.

The glovebox will then be sealed and verified to be at negative pressure relative to the room pressure. The TCs will be opened using a pressure relief device (PRD) that allows the TC plug to be removed while controlling the release of any pressure that may exist in the TC.

After opening the "working" plug, liquid samples will be collected using new tubing placed through the TC plug hole and into the agent. For GA and Lewisite TCs 0.50 mL each will be taken from the 25%, 50%, and 75% agent fill levels (horizontal TC orientation) and composited to form one (1) 1.5 mL sample. If a heel is present, a 1.5 mL sample will also be taken. The "transparency" TC will be head space monitored for Lewisite using a MINICAMS and a liquid sample (1.5 mL) taken from the TC if liquid is present. The samples will be transferred to vials (≤ 5 mL vials), lids sealed on the vials, vials placed in overpacks, and transferred to the glovebox airlocks. After each sample is collected, the sampling equipment (syringe and tubing) will be placed into the TC through the TC plug hole. Information regarding each TC and sample collected will be recorded on a Glovebox Operations Worksheet.

After sampling has been completed, the overpacked samples will be removed from the glovebox airlocks and placed in containers and overpacked for shipment to the HMRC. Form DD-1911 will be completed to document chain of custody.

7. Objective four will not be met using this sampling plan. Additional waste characterization of these ton containers will be needed.

We requested that DSHW provide us a better understanding behind the intent of this comment. DSHW stated that we should not read these words literally, their intent was to make the point that they cannot commit to the data supporting "no additional analysis" until they are able to review the results and the associated quality control data. They may still require additional verification sampling during shakedown, etc., depending on the analytical results.

Resolution TOCDF can accept this position and will follow up with DSHW after submittal of the analytical results to DSHW for their review, and as part of the Class 3 Permit Modification process

- 8 Analytical SOPs that are not in SW-846 or permit approved methods will need to be submitted for Board approval

DSHW has retracted this comment, the SOPs do not require submittal to the Board for approval – we will, however, need to work with DSHW on any such methods

SOPs for the activities associated with this effort have been included in the sampling plan appendices (1-4)

- 9 Section 3 1 3 Collection of GA/Lewisite Samples Composite samples should also be fabricated from aliquots collected from various depths in the GA ton container as will be done for the Lewisite ton containers

DSHW indicated that as we discussed in our response to comment #2, composite samples are more representative of waste feed

Resolution TOCDF indicated that composite sampling could be performed for the GA TCs

This comment has been addressed in Section 3 1 3 as follows

“After opening the “working” plug, liquid samples will be collected using new tubing placed through the TC plug hole and into the agent For GA and Lewisite TCs 0 50 mL each will be taken from the 25% 50% and 75% agent fill levels (horizontal TC orientation) and composited to form one (1) 1 5 mL sample ”

- 10 How will CARA WEST manage their wastes, such as air filters, PPE, sampling tools etc ? Where is the waste management plan for HMRC that they are proposing to follow

We need to develop more details in coordination with CARA West – DSHW has no specific concerns, but wants to make sure they understand how the waste will be managed The HMRC waste management plans can be provided to DSHW

Resolution TOCDF committed to providing DSHW with CARA West's and HMRC's waste management plans

Waste disposition has been addressed in Section 3 1 8 as follows

“Waste generated by CARA West during the sampling operation will be stored by DCD and disposed of by TOCDF during the CAMDS/DCD Secondary Waste campaign Sample and analytical residues will be managed according to the HMRC waste disposal plans ”

- 11 Does CARA WEST have calibration procedures for all sampling equipment, such as thermometers?

We need to coordinate with CARA West and obtain more details on the instruments they intend on using and any appropriate calibration procedures being used, the information will be incorporated in the Plan or communicated to DSHW

SOPs for the activities associated with this effort have been included in the sampling plan Appendices (1-4)

- 12 Section 3 1 5 Why are you using Quantitative rather than Qualitative methods?

Note that in the discussions with DSHW, TOCDF clarified that the intent of the comment was to question the use of qualitative (vs quantitative) methods This comment was addressed during the discussion of comment #4 and #5

Resolution DSHW is satisfied with TOCDF's responses with respect to the manner in which quantitative methods are being utilized for this sampling effort

13 What are the impurities for GA and Lewisite that will be analyzed for?

See response to comment #4

Resolution DSHW would like us to include a more thorough description in the Plan of the analytical/TIC identification process

This comment has been addressed in Section 3 2 2, "Agent purity and TICs for Lewisite liquid samples" as follows

"Analysis will be performed by GC/MS per SOP HMRC IV-055 to determine the retention time (RT) of Lewisite 1 and 2, identified as their thioether derivatives, and Lewisite 3. A NIST mass spectral library will be used to evaluate samples for TICs. Reports will be generated using ChemStation software."

14 Section 3 2 2 Why will Density not be performed on Lewisite sludge samples?

Density information is not required to support treatment

Resolution DSHW accepted TOCDF's rationale

15 Section 4 3 2 Please use 'less than' values rather than "Not Detected" to report

Resolution TOCDF will make this change to the Plan

This comment has been addressed in Section 4 3 2 as follows

"Data for analytes that are not detected will use the EQL for the lower reporting limit, and will be reported using "less than" values."

16 A phase determination should include liquid and non-liquid/ sludge

(See response to comment #2 with respect to "phase determination", DSHW requested that TOCDF consider having the samplers and/or laboratory personnel document a physical description of the sample and note if the presence of multiple phases were observed)

Resolution TOCDF will work with CARA West and HMRC to satisfy DSHW's request

This comment has been addressed in Section 3 1 7 as follows

"At the HMRC, the samples will be received, logged and stored by trained authorized personnel according to Department of Army regulations. A visual observation of each sample will be made and the results recorded to capture the color, consistency, and number of phases for each sample. Samples will be stored at 4-6°C upon receipt at the HMRC."

17 How are the samples digested for metals at HMRC, and why are they then shipped back to TOCDF instead of analyzing them with the other parameters at HMRC?

TOCDF personnel explained the rationale for this, which was based upon equipment and method availability

Resolution DSHW was satisfied with TOCDF's rationale/response

This comment has been addressed in Section 3 2 2 HRA Metals for GA, Lewisite liquid, Lewisite sludge, and "transparency" ton liquid samples as follows

"Samples will be prepared (digested) at the HMRC and analyzed at Battelle's laboratory at King Ave., Columbus, OH. Sample preparation and analysis will follow a project specific SOP based on TE-LOP-557 and TE-LOP-584. Samples will be confirmed below research, development, testing and evaluation (RDTE) dilute solution limits for GA and L prior to shipment to King Ave. Transparency ton liquid samples will be treated in the same manner as the liquid and sludge samples and analyzed on a weight basis. The EQLs for the target metal analytes are summarized in Table 1. Actual EQLs for the

samples will be dependent on the sample weight, preparation dilution factor, and density ”

- 18 Sample duplicate should be clarified that it is a split of a field sample performed at the preparation/analytical laboratory

Resolution TOCDF will make this change to the Plan

This comment has been addressed in Section 4.1.1 as follows

“Sample Duplicate – A split of a field sample (generated at the analytical laboratory) used to provide estimates of precision for sample results that are >5X EQL ”

- 19 Samples that do not meet the acceptability criteria may be re-extracted/re-analyzed if the holding time has not been exceeded

Resolution TOCDF will make this change to the Plan

This comment has been addressed in Section 4.2 as follows

“A summary of analytical quality requirements for the sampling program is presented in Tables 3 through 9. QC results outside of the control limits will be addressed as outlined in Tables 3 through 9. Samples that do not meet QC acceptability criteria may be re-extracted/re-analyzed if sample holding times have not been exceeded ”

- 20 Mercury samples must be kept at 4-6°C for holding time

This issue has been addressed in the past in support of previous agent characterization efforts. Based on a stability study for Hg in mustard, and the fact that the agent samples will be sealed such that Hg vapors should not escape, we do not believe that this temperature requirement has any technical validity.

Resolution DSHW requested TOCDF to verify that the proposed approach is consistent with what was done for HD. DSHW acknowledged that they had agreed to this resolution in the past and indicated that they would not impose this temperature requirement for the time periods in transit, when the agent samples are contained within sealed vials. DSHW's expectation is that the temperature requirement will be maintained at the receiving laboratory per Battelle procedures.

- 21 Table 4 Sample duplicate criteria is too wide, should be 5%

Resolution TOCDF will make this change to the Plan

Table 4 has been updated to reflect this change

- 22 Table 5 Balance should have sensitivity check also

Resolution TOCDF will make this change to the Plan

Table 5 has been updated to reflect this change

- 23 Table 6 Initial calibration and CCV should not be NONE. Need to include impurities

TOCDF personnel explained the method in question utilizes a GC/FID detector, which provides information about the content of a solution relative to that solution only (compares the sample to itself using relative peak area to quantitate the sample components), therefore external calibration is not required.

Resolution DSHW accepted TOCDF's rationale/response

- 24 Why is the blank spike and matrix spike the same criteria. The blank spike range should be tighter

Resolution TOCDF will evaluate and address as appropriate

Blank and matrix spike criteria are similar because the analysis is by FID detector, which determines concentration as relative peak area and does not rely on external calibration for quantification

25 Table 8 Instrument Tune in the Quality Parameter column should be every 12 hours in the Method/Frequency column, also should have an initial calibration and CCV checks in the table
The instrument will be tuned as appropriate The response to the second part of this comment (calibration and CCV checks) was addressed during the discussion on comment #23
Resolution DSHW accepted TOCDF's rationale/response

26 Table 9 Needs to include MS/MSD with sample requirements
Resolution DSHW indicated this comment was no longer applicable, based on prior discussions (see comment #23)

27 Will the Division be notified of any deviation from the sampling plan?
Yes

Resolution TOCDF will make a change to the Plan to specify this

The Plan text has been amended in Section 1.4 to address this concern as follows

“The Utah Department of Environmental Quality, Division of Solid and Hazardous Waste will be notified of any changes/deviations to this sampling plan “

GA/Lewisite Disposal Facility

Ton Container Sampling Program Plan

**June 1, 2009
Revision 1**

1	6/1/09	Incorporated changes based on Utah DSHW comments regarding Revision 0 Added TIC analysis parameters for GA and L liquids, updated metals EQLs, added Tables 10 and 11, added ECBC-TR-531 reference method for the analysis of L1, L2, and L3	AES/TAM
0	2/26/09	Initial release	MDS
Rev	Date	Reason for Revision	By

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1 INTRODUCTION

1.1 Background

The GA, GA/UCON and Lewisite munitions stored at Deseret Chemical Depot (DCD) include ten (10) ton containers (TCs) of Lewisite, two (2) TCs of GA and two (2) GA/UCON TCs. Additionally there are ten 10 ton containers considered "empty" that have been declared under the "transparency" provision of the OPCW treaty.

Two (2) GA ton containers have approximately 2,821 pounds of agent GA, while the GA/UCON ton containers have approximately 1,286 pounds of agent GA. The history and previous analysis of the GA/UCON ton containers indicate that these TCs and the agent in them were prepared for the addition of UCON, a poly-glycol thickening agent, but due to difficulties with the test apparatus, UCON was never added.

The ten (10) TCs of Lewisite contain an aggregate mass of approximately 13 tons of agent Lewisite. The ten (10) "transparency" TCs are believed to have once contained Lewisite and have been cleaned to various levels of decontamination, some of these TCs may have already been decontaminated and monitored to < 10 vapor screening limit (VSL). A portion (five) of the "transparency" TCs have been identified as part of a group, that while stored at Pine Bluff Arsenal, were cleaned and rented to industry.

The GA/Lewisite Sampling Program will be conducted by CARA West (formerly the 22nd Infantry Chemical Battalion) in Area 10 of the Deseret Chemical Depot (DCD). The general approach involves receipt of GA/Lewisite TCs from Area 10 personnel, inspection of TCs for leaks or scabs, and verification of the TC "D" numbers. CARA West personnel will prepare the TCs for sampling by placing them in a glovebox for the collection of samples from the TCs. After sampling, the TCs will be deconned, monitored and returned to storage in Area 10.

1.2 Objectives

Objective 1 To collect liquid samples at the 25%, 50%, and 75% fill levels (horizontal TC orientation) and composite these samples into a single liquid sample for each of the GA TCs. If present, a sludge sample will be collected from each of the GA TCs with effort taken to ensure that only sludge (no liquid) is collected.

- The GA will be analyzed for agent purity, density, pH, Health Risk Assessment (HRA) metals, chlorobenzene content, and tentatively identified compounds (TICs) as identified by GC/MS.
- Each sludge sample, if present, will be analyzed for HRA metals.

Objective 2 To collect liquid samples at the 25%, 50%, and 75% fill levels (horizontal TC orientation) and composite these samples into a single liquid sample for each of the Lewisite TCs. If present, a sludge sample will be collected from each of the Lewisite TCs with effort taken to ensure that only sludge (no liquid) is collected.

- Each liquid Lewisite sample will be analyzed for agent purity (L1, L2, L3), density, pH, HRA metals, and tentatively identified compounds (TICs) as identified by GC/MS.

- Each sludge sample, if present, will be analyzed for HRA metals

Objective 3 To monitor the headspace of the “transparency” TC for Lewisite using MINICAMS® and for volatile organic compounds using a sorbent tube (only collected if Lewisite not detected in the headspace using MINICAMS) If present, a liquid sample will be collected

- The results of the head space sampling will determine if the TCs will be handled the same as the drained Lewisite TCs
- Liquid samples will be analyzed for L1 (quantitative), L2 and L3 (qualitative), HRA metals (quantitative), and TICs as identified by GC/MS

Objective 4 It is intended that this sampling and analysis effort will sufficiently characterize GA/Lewisite agent feed to the Area 10 liquid incinerator in a manner that is acceptable to the State of Utah, Division of Solid and Hazardous Waste

Objective 5 The overall quality objective is to ensure generation of analytical data that may be used to give insight to the problems that might arise in the incineration of the agent and the clearing of the TCs for off-site disposal

1 3 Organization

The Chemical Materials Agency (CMA), DCD, and EG&G Defense Materials (EG&G) have shared interest and responsibilities for this sampling program CMA has oversight responsibility of the program DCD will allow CARA West operator’s access to the Area 10 to conduct sampling operations DCD will deliver TCs to the igloos for sampling, and will return the TCs into storage after sampling CARA West will perform all headspace monitoring, collect all agent samples, and seal and package the agent samples for transport to the Battelle Hazardous Materials Research Center (HMRC) for analysis EG&G or its contractors will perform all other actions associated with this program

The EG&G Area 10/Secondary Waste Operations Manager has overall Project Management responsibility for the sampling program

1 4 Changes to the Plan

To ensure that test objectives and quality standards are met, this plan will be implemented as written Only the EG&G Area 10/Secondary Waste Operations Manager or his/her designee may approve deviations/changes to this plan

The Utah Department of Environmental Quality, Division of Solid and Hazardous Waste will be notified of any changes/deviations to this sampling plan

2 SAFETY CRITERIA

All work will be performed in accordance with (IAW) applicable U S Army regulations. In addition, samples will be analyzed IAW applicable Standard Operating Procedures (SOPs) and Laboratory Operating Procedures (LOPs).

3 SAMPLING STRATEGY

3.1 Sampling Objectives

The objectives of the GA/Lewisite Sampling Program are to collect representative samples from the GA, GA/UCON, Lewisite and "transparency" TCs to support the processing in a liquid incinerator system to be constructed in Area 10 of the DCD. The results of the analysis will allow engineering plans and controls to be added to the planned disposal system to aid the incineration of the agent and the clearing of the TCs for off-site disposal. It is intended that this sampling and analysis effort will sufficiently characterize GA/L agent feed to the liquid incinerator in a manner that is acceptable to the State of Utah, Division of Solid and Hazardous Waste.

3.1.1 Statistical Objective

The statistical objective of this program is to obtain representative and defensible analytical results for the two (2) GA TCs, the two (2) GA/UCON TCs, the ten (10) Lewisite TCs and the ten (10) "transparency" TCs currently stored at DCD.

3.1.2 Sampling Accuracy

Sampling accuracy will be achieved by sampling each GA, GA/UCON, Lewisite, and "transparency" TC in a consistent manner as defined by CARA West SOP TU-0000-M-076.

3.1.3 Collection of GA/Lewisite Samples

EG&G will notify DSHW at least 72 hours in advance of the initiation of Area 10 GA/Lewisite sampling operations. DCD will notify OPCW as required. Sample collection, packaging and shipping to the HMRC will be accomplished by CARA West IAW Army Regulations.

CARA West will arrive with their specialized equipment consisting of glove boxes, air filters, generators, decontamination facilities, agent monitoring, and other supplies necessary to collect the agent samples to complete the sampling program. Air monitoring stations will be established using instruments operating under existing CARA WEST Precision and Accuracy studies. CARA West will perform alternate baseline monitoring studies (Per CMA LMQAP Section 10.4) while on-site and submit them to CMA for review and concurrence.

After confirmation that the glove boxes are working properly, the GA, GA/UCON, Lewisite, and "transparency" TCs will be received from Area 10 and placed in the glovebox. The TCs will be positioned such that the heel weather mark is toward the bottom (horizontal TC orientation), the

storage arrow marked by Area 10 is visible at the top, and the “working” plug is at the top. Preliminary physical examination of the TCs (5/19/09) suggests the presence/absence of heel material for the TCs. This information will ensure that the sampling effort is conducted efficiently.

Prior to placement in the glovebox, the CARA West Sampling Operators will measure and record the external temperature of each TC.

The glovebox will then be sealed and verified to be at negative pressure relative to the room pressure. The TCs will be opened using a pressure relief device (PRD) that allows the TC plug to be removed while controlling the release of any pressure that may exist in the TC.

After opening the “working” plug, liquid samples will be collected using new tubing placed through the TC plug hole and into the agent. For GA and Lewisite TCs 0.50-mL each will be taken from the 25%, 50%, and 75% agent fill levels (horizontal TC orientation) and composited to form one (1) 1.5-mL sample. If a heel is present, a 1.5-mL sample will also be taken. The “transparency” TC will be headspace monitored for Lewisite using a MINICAMS and a liquid sample (1.5-mL) taken from the TC if liquid is present. The samples will be transferred to vials (≤ 5 -mL vials), lids sealed on the vials, vials placed in overpacks, and transferred to the glovebox airlocks. After each sample is collected, the sampling equipment will be placed into the TC through the TC plug hole. Information regarding each TC and sample collected will be recorded on a Glovebox Operations Worksheet.

After sampling has been completed, the overpacked samples will be removed from the glovebox airlocks and placed in containers and overpacked for shipment to the HMRC. Form DD 1911 will be completed to document chain of custody.

3.1.4 Headspace Lewisite monitoring for “transparency” TCs

Monitor “transparency” ton containers for L1 only using MINICAMS with a Lewisite derivatization module and a halogen specific detector (XSD). No confirmation analysis will be performed if Lewisite is detected.

3.1.5 Headspace volatile organic compounds (VOCs) for “transparency” TCs

Solid sorbent tubes will be used to collect samples from ton container headspace for qualitative VOC identification and semi-quantitative analysis.

3.1.6 Sample Transport

CARA West personnel will transport the overpacked sample containers to the HMRC for analysis.

3 1 7 Sample Receipt and Storage

At the HMRC, the samples will be received, logged and stored by trained authorized personnel according to Department of Army regulations. A visual observation of each sample will be made and the results recorded to capture the color, consistency, and number of phases for each sample. Samples will be stored at 4-6°C upon receipt at the HMRC.

3 1 8 Sample and Analytical Waste Disposal

Waste generated by CARA West during the sampling operation will be stored by DCD and disposed by TOCDF during the CAMDS/DCD Secondary Waste campaign. Sample and analytical residues will be managed according to the HMRC waste disposal plans.

3 1 9 Sampling Records

Collection of each liquid GA, GA/UCON, Lewisite, and "Transparency" TC sample will be documented by a Glovebox Operations Worksheet. Information collected will as a minimum include the following:

- Operator Name
- Sample Collection Date
- Igloo Number
- Glovebox Number
- TC "D" Number
- Sample Collection Time
- Sample ID Number
- TC Temperature
- Agent type (GA, L, Transparency)
- Quantity of sample
- Physical Appearance (Color, Phase(s), Viscosity)

3 2 Analytical Procedures

3 2 1 Applicable Quality Assurance Program Plans

The TOCDF Participant Quality Assurance Plan (PQAP) (CDRL 22), the TOCDF Laboratory Quality Control Plan (LQCP), and the Battelle Chemical Environmental and Materials Operations (CEMO) Quality Manual will be followed for this program. The revisions of these documents current at the time of sample collection and analysis will apply.

3 2 2 Laboratory Operating Procedures

The liquid, sludge and associated QC samples will be prepared, analyzed, and reported IAW the following methods. Revisions of these documents current at the time of sample analysis will apply.

pH for GA, Lewisite liquid, and “transparency” TC liquid samples

pH will be determined following EPA Method 9045D using a pH meter. A 10 g sample will be used instead of the specified 20 g sample. No Estimated Quantitation Limit (EQL) for this method.

Density for GA and Lewisite liquid samples

A Class A syringe will be used to deposit a known volume of agent onto a balance capable of measuring to 0.1 mg. Balance will be calibrated with NIST traceable weights. Measurement will be performed at ambient temperature. Density will be determined as measured mass divided by measured volume. Density will not be performed on Lewisite sludge samples. No EQL for this method.

Agent purity for GA liquid samples

Purity will be determined following SOP HMRC IV-055 “Purity and Impurity Analysis of Solutions Containing Chemical Agent by Gas Chromatography”. Purity will be reported as a percentage on a mass basis. No EQL for this method.

Tentatively identified compounds (TICs) for GA liquid samples

TICs will be determined following SOP HMRC IV-055. Samples will be analyzed by full scan GC/MS. A NIST mass spectral library will be used to evaluate samples for TICs. Reports will be generated using ChemStation software.

Chlorobenzene (CAS # 108-90-7) for GA liquid samples

GA liquid samples will be analyzed by GC/MS following guidelines of SOP HMRC-056 “Operation and Maintenance of Gas Chromatography and Analysis of Solutions Containing GA, GB, GD, GF, HD, L, and VX by Gas Chromatography”. EQL of 1 mg/mL based on 10 µL sample diluted in 10 mL of solvent with a low calibration point of 10 µg/mL.

Agent purity and TICs for Lewisite liquid samples

Samples will be dissolved in acetone and derivatized with 1-propane thiol or other appropriate thiol. Analysis will be performed by GC/MS per SOP HMRC IV-055 to determine the retention time (RT) of Lewisite 1 and 2, identified as their thioether derivatives, and Lewisite 3. A NIST mass spectral library will be used to evaluate samples for TICs. Reports will be generated using ChemStation software. GC/MS RT data will be applied to purity analysis by GC/FID per SOP HMRC IV-055. Purity will be reported as a percentage on a mass basis. No EQL for this method.

L1, L2 and L3 for “transparency” ton liquid samples

Samples will be prepared and analyzed in accordance with the procedures reported in ECBC-TR-531 for the quantitative analysis of L1, L2 and L3. GC/MS calibration standards will be prepared from Lewisite stock available at the HMRC. The MDL reported for L1 by this method is 25 µg/L. The estimated quantitation limit for a 0.5 mL sample extracted into 5.0 mL of 2,2,4-trimethylpentane is 150 µg/L. Direct quantification of L2 and L3 may not be possible, however these compounds will be identified based on the spectral identification of their components.

Headspace volatile organic compounds (VOCs) for “transparency” ton sorbent tube samples

Solid sorbent tubes samples collected from ton container headspace for qualitative VOC identification and semi-quantitative analysis will be thermally desorbed and analyzed by GC/MS following guidelines of SOP HMRC-067 “Quantitative Analysis of HD, GA, GB, GD, and VX Collected on Solid Sorbent”. Detected components will be qualitatively identified by mass spectral library match. Semi-quantitative results will be reported by comparison to an internal standard. No EQL for this method.

HRA Metals for GA, Lewisite liquid, Lewisite sludge, and “transparency” ton liquid samples

Samples will be prepared (digested) at the HMRC and analyzed at Battelle’s laboratory at King Ave., Columbus, OH. Sample preparation and analysis will follow a project specific SOP based on TE-LOP-557 and TE-LOP-584. Samples will be confirmed below research, development, testing and evaluation (RDTE) dilute solution limits for GA and L prior to shipment to King Ave. Transparency ton liquid samples will be treated in the same manner as the liquid and sludge samples and analyzed on a weight basis. The EQLs for the target metal analytes are summarized in Table 1. Actual EQLs for the samples will be dependent on the sample weight, preparation dilution factor, and density.

3.2.3 Analytical Samples

The HMRC will be responsible for the preparation of QC samples and analysis of all samples collected in Area 10. Field duplicate samples will be collected at a frequency of one per TC type, e.g., (1) GA, GA/UCON, (2) Lewisite, and (3) “transparency”, for liquid samples only.

Laboratory QC samples are shown in Section 4.1.1. The HMRC will prepare and analyze laboratory QC samples at the frequencies outlined in the tables in Section 4.

3.2.4 Estimated Quantitation Limits (EQLs)

The EQLs for the target analytes are summarized in Tables 1 and 2. Actual EQLs for the samples will be dependent on the sample weight, preparation dilution factor, and sample density.

3.3 Instrument Calibration

Prior to analysis, instrument calibration status will be verified IAW the applicable procedure. If necessary, instruments will be calibrated (or recalibrated).

Table 1
EQLs for Target Analytes

Analyte	Estimated Quantitation Limit ¹
pH	Not Applicable
Density	Not Applicable
Agent Purity	Not Applicable
Chlorobenzene (GA TCs)	1 mg/mL
L1 ("transparency" TCs)	150 µg/L
L2, L3 ("transparency" TCs)	Qualitative Identification
Headspace VOCs ("transparency" TCs)	Not Applicable
¹ These are approximate EQL values. The actual EQLs for the samples are dependent on the sample weight, preparation dilution factor, and actual sample density.	

Table 2
EQLs for Target Metal Analytes

Metal	EQL¹ (mg/kg)
Aluminum	5
Antimony	0.05
Arsenic	0.05
Barium	0.05
Beryllium	0.05
Boron	5
Cadmium	0.05
Chromium	0.05
Cobalt	0.05
Copper	0.05
Lead	0.05
Manganese	0.05
Mercury	0.25
Nickel	0.05
Selenium	1.25
Silver	0.5
Thallium	0.05
Tin	0.05
Vanadium	0.05
Zinc	5

¹ Estimated Quantitation Limits (EQLs) based on a 0.2 g sample size and 100 mL final sample volume

4 QUALITY ASSURANCE AND REPORTING

4.1 Quality Control Objective

The overall quality control objective is to ensure generation of accurate analytical data that may be used to give insight to the problems that might arise in the incineration of the agent and the clearing of the TCs for off-site disposal.

4.1.1 QC Samples

The types of QC samples that will be used to document the validity of the data generated from this program are described below. Specific QC samples for each test are listed in Tables 3 through 9.

- Continuing Calibration Blank (CCB) – A standard solution that contains the same acids or solvent and concentrations and the same internal standards and concentrations as the calibration standards.

- **Initial Calibration Verification (ICV)** – A standard solution analyzed to demonstrate the initial instrument calibration
- **Continuing Calibration Verification (CCV)** – A standard solution analyzed to demonstrate the instrument calibration remains acceptable
- **Method (Preparation) Blank** – Acids and other reagents used during sample preparation that are carried through the entire sample preparation and analysis method using the same reagents and volumes as is done for the samples
- **Reagent Blank** – A sample containing all components, except the analyte, that is carried through all steps of the analysis method, except for sample preparation
- **Blank Spike** – A sample consisting of all reagents and has been spiked with a known quantity of the target analyte(s)
- **Dilution Test (Applicable to HRA metals analysis only)** – Dilution test results will be evaluated if the analyte concentration is within the linear dynamic range of the instrument and is greater than 100 times the instrument detection limit (IDL) for any target analyte. An analysis of a fivefold (1+4) dilution must agree to within $\pm 10\%$ of the original determination. If not, an interference effect must be suspected.
- **Post-digestion Spike (PDS) (Applicable to HRA metals analysis only)** – An analyte spike added to a portion of a prepared sample, or its dilution, that should be recovered to within 75% to 125% of the known value or within the laboratory derived acceptance criteria
- **Laboratory Control Sample (LCS)** - The LCS is carried through the entire procedure from sample preparation through analysis as if it were a field sample. The purpose of the LCS is to evaluate bias of the method
- **Matrix Spike (MS)** – An aliquot of sample spiked with a known concentration of target analytes. The spiking occurs prior to sample preparation and analysis. Used to document the precision and bias of a method in a given matrix
- **Matrix Spike Duplicate (MSD)** – Intralaboratory split samples spiked with identical concentrations of target analytes. The spiking occurs prior to sample preparation and analysis. Used to document the precision and bias of a method in a given matrix
- **Sample Duplicate** – A split of a field sample (generated at the analytical laboratory) used to provide estimates of precision for sample results that are $>5X$ EQL
- **Field Duplicate** – Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process

4.2 QC Requirements

A summary of analytical quality requirements for the sampling program is presented in Tables 3 through 9. QC results outside of the control limits will be addressed as outlined in Tables 3 through 9. Samples that do not meet QC acceptability criteria may be re-extracted/re-analyzed if sample holding times have not been exceeded.

4.2.1 Precision

Precision is defined as the degree of mutual agreement among individual measurements made under prescribed conditions. The precision goals are included in Tables 3 through 9.

Precision will be calculated for laboratory duplicate analysis using the following equation:

$$RPD = \left[\frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2} \right)} \right] \times 100$$

Where

- RPD = Relative Percent Difference
- X_1 = Analytical Result of Sample
- X_2 = Analytical Result of Duplicate

4.2.2 Accuracy

Accuracy is the degree of agreement of a measurement to an accepted reference or true value. The accuracy will be determined from analysis of samples spiked with a known concentration. Accuracy objectives have been set and are presented in Tables 3 through 9. The formula which will be used to assess the accuracy of the laboratory QA/QC data (e.g., matrix spike analysis) is as follows:

$$\%R = \left(\frac{(Q_{ss} - Q_{us})}{Q_s} \right) \times 100$$

Where

- $\%R$ = Percent recovery
- Q_{ss} = Quantity of Analyte Found in the Spiked Sample
- Q_{us} = Quantity of Analyte Found in the Unspiked Sample
- Q_s = Quantity of Added Spike

Note: For ICV/CCV, no unspiked samples, therefore $Q_{us} = 0$

4 2 3 Completeness

Completeness is defined as the amount of valid data from a measurement system compared to the amount that was expected under optimal normal conditions. Completeness should be 100%.

While only a single 1.5 – 2.0 mL sample will be collected from each TC, not all of the sample will be consumed during analysis. If the results of the initial analysis are not acceptable for any reason, the remaining portion of the sample will be analyzed, except for pH testing, which will be performed in duplicate, and VOCs, for which there will only be one sample collected.

Completeness will be reported as the percentage of all measurements judged to be valid. Every attempt will be made to ensure that all data generated will be valid data. If data appears questionable based on circumstances that occurred or were observed during either the field sampling or laboratory analyses (i.e., sampling or analytical methods were not followed, unreasonable results, or equipment), it will be flagged and an explanation provided.

Table 3
QA/QC Criteria for Determination of HRA Metals

Quality Parameter	Method/Frequency	Criteria¹
ICS	Beginning of analytical run or once every 12 hours, whichever is more frequent	70% to 130% recovery
CCB	At least once every 10 analytical samples	Response < 3 x IDL
CCV	At least once every 10 analytical samples	Results \pm 10% of initial calibration
Preparation Blank	At least once every 20 analytical samples	< EQL (PQL)
Dilution Test	At least once every 20 analytical samples	Within \pm 10% of original determination where elements are found within the linear calibration range of the instrument for both the parent and the dilution
PDS	At least once every 20 analytical samples	75% to 125% recovery
LCS	At least once every 20 analytical samples	\pm 25% of spike amount
MS	At least once every 20 analytical samples	\pm 25% recovery
MSD	At least once every 20 analytical samples	\leq 20% RPD
Sample Duplicate	At least once every 20 analytical samples	\leq 20% RPD if response >100 IDL
Field Duplicate	One per TC type, e g , (1) GA, GA/UCON, (2) Lewisite, and (3) "transparency", for liquid samples only	\leq 25% RPD if response >100 IDL
Holding Time	Every sample	28 Days (Mercury) 6 Months (All other metals)

¹ Corrective actions will be captured in the project specific SOP

Table 4
QA/QC Criteria for Determination of pH for GA and L

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Initial Calibration	Daily prior to use		
Continuing Calibration Checks	Every 2 hr		
Internal Standards	None		
MS/MSD	None		
Sample Duplicate	1 per sample	$RPD \leq 5\%$	Check Calculations, Assess Impact on Data, Narrate
Blank Spike	None		
Method Blank	None		
Reagent Blank	1 per sample		
Holding Time		60 days	

Table 5
QA/QC Criteria for Determination of Density for GA and L

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Initial Calibration	Daily prior to use, to include sensitivity check (balance)		
Continuing Calibration Checks	None		
Internal Standards	None		
MS/MSD	None		
Sample Duplicate	1 per sample	$RPD \leq 10\%$	Check Calculations, Assess Impact on Data, Narrate
Blank Spike	None		
Method Blank	1 per batch 20 samples using deionized water		
Reagent Blank	None		
Holding Time		60 days	

Table 6
QA/QC Criteria for Determination of GA purity by GC/FID

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Initial Calibration	None		
Continuing Calibration Checks	None		
Internal Standards	None		
MS/MSD	None		
Sample Duplicate	1 per Batch (Every 20 Samples)	$RPD \leq 25\%$	Check Calculations, Assess Impact on Data, Narrate
Blank Spike	None		
Method Blank	None		
Reagent Blank	1 per sample	Used to baseline correct sample response	
Holding Time		60 days	

Table 7
QA/QC Criteria for Determination of chlorobenzene in GA by GC/MS

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Instrument Tune (PFTBA)	Initially and as required	Method Key Ion and Ion Abundance Criteria	Repeat Instrument Tune
Instrument Evaluation and Performance Check (50 µg/ml DFTPP)	Initially, Daily, and as Required	EPA Method 8270C DFTPP Criteria	Evaluate System, Perform Corrective Maintenance Recalibrate
Initial Calibration	5 point calibration of CLB	$R^2 \geq 0.990$ Each Level $\pm 15\%$ of Theoretical Value	Evaluate System Recalibrate System
Continuing Calibration Checks	Low calibration standard concentration and second-highest calibration standard concentration, independently prepared, Every 5 Samples	Within $\pm 25\%$	Reanalyze If outside $\pm 25\%$, Perform maintenance on Instrument Recalibrate Reanalyze Affected Samples
Internal Standards	RT	Within ± 30 Seconds of Continuing RT Value	Check Sensitivity of System Reanalyze samples or standards
MS/MSD	1 per Analytical Batch (Every 20 Samples)	Recovery 50 to 150% RPD $\leq 25\%$	Check Calculations, Assess Impact on Data, Narrate
Sample Duplicate	1 per Batch (Every 20 Samples)	RPD $\leq 25\%$	Check Calculations, Assess Impact on Data, Narrate
Blank Spike	1 per Analytical Batch (Every 20 Samples)	Recovery 50 to 150%	Check Calculations, Assess Impact on Data, Narrate
Method Blank	1 per Analytical Batch (Every 20 Samples)	$< \text{EQL (PQL)}$	Reanalyze if analysis $\geq \text{EQL (PQL)}$
Reagent Blank	1 per Analytical Batch (Every 20 Samples)	$< \text{EQL (PQL)}$	Reanalyze if analysis $\geq \text{EQL (PQL)}$
Holding Time		60 days	

Table 8
QA/QC Criteria for Determination of L purity by GC/MS and GC/FID

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Instrument Tune (PFTBA)	Initially and as required	Method Key Ion and Ion Abundance Criteria	Repeat Instrument Tune,
Instrument Evaluation and Performance Check (50 µg/ml DFTPP)	Initially, Daily, and as Required	EPA Method 8270C DFTPP Criteria	Evaluate System, Perform Corrective Maintenance Recalibrate
Initial Calibration	None		
Continuing Calibration Checks	None		
Internal Standards	None		
MS/MSD	None		
Sample Duplicate	1 per Batch (Every 20 Samples)	RPD ≤ 25%	Check Calculations, Assess Impact on Data, Narrate
Blank Spike	None		
Method Blank	None		
Reagent Blank	1 per sample	Used to baseline correct GC/FID sample response	
Holding Time		60 days	

Table 9
QA/QC Criteria for Determination of unknown components in TC headspace by GC/MS

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Instrument Tune (PFTBA)	Initially and as required	Method Key Ion and Ion Abundance Criteria	Repeat Instrument Tune
Instrument Evaluation and Performance Check (50 µg/ml BFB)	Initially, Daily, and as Required	EPA Method 8260C BFB Criteria	Evaluate System, Perform Corrective Maintenance Recalibrate
Initial Calibration	None		
Continuing Calibration Checks	None		
Internal Standards	RT	Within ±30 Seconds of Continuing RT Value	Check Sensitivity of System
MS/MSD	None		
Sample Duplicate	None		
Blank Spike	None		
Method Blank	1 per Analytical Batch (Every 20 Samples)	Baseline comparison to sample data	
Reagent Blank	None		
Holding Time		28 days	

Table 10
QA/QC Criteria for Determination of TICs in GA and L Liquids by GC/MS

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Instrument Tune (PFTBA)	Initially and as required	Method Key Ion and Ion Abundance Criteria	Repeat Instrument Tune
Instrument Evaluation and Performance Check (50 µg/ml DFTPP)	Initially, Daily, and as Required	EPA Method 8270C DFTPP Criteria	Evaluate System, Perform Corrective Maintenance Recalibrate
Initial Calibration	None		
Continuing Calibration Checks	None		
Internal Standards	None		
MS/MSD	None		
Sample Duplicate	1 per Batch (Every 20 Samples)	Qualitative comparison between samples	
Blank Spike	None		
Method Blank	None		
Reagent Blank	1 per sample	Baseline comparison to sample data	
Holding Time		60 days	

Table 11
QA/QC Criteria for Determination of L1, L2 and L3 in transparency ton liquid samples by GC/MS

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Instrument Tune (PFTBA)	Initially and as required	Method Key Ion and Ion Abundance Criteria	Repeat Instrument Tune
Instrument Evaluation and Performance Check (50 µg/ml DFTPP)	Initially, Daily, and as Required	EPA Method 8270C DFTPP Criteria	Evaluate System, Perform Corrective Maintenance Recalibrate
Initial Calibration	5 point calibration of L1	$R^2 \geq 0.990$ Each Level $\pm 15\%$ of Theoretical Value	Evaluate System Recalibrate System
Continuing Calibration Checks	Low calibration standard concentration and second-highest calibration standard concentration, independently prepared, Every 5 Samples	Within $\pm 25\%$	Reanalyze If outside $\pm 25\%$, Perform maintenance on Instrument Recalibrate Reanalyze Affected Samples
Internal Standards	None		
MS/MSD	1 per Analytical Batch (Every 20 Samples)	Recovery 50 to 150% $RPD \leq 25\%$	Check Calculations, Assess Impact on Data, Narrate
Sample Duplicate	1 per Batch (Every 20 Samples)	$RPD \leq 25\%$	Check Calculations, Assess Impact on Data, Narrate
Blank Spike	1 per Analytical Batch (Every 20 Samples)	Recovery 50 to 150%	Check Calculations, Assess Impact on Data, Narrate
Method Blank	1 per Analytical Batch (Every 20 Samples)	$< L1 \text{ EQL}$	Reanalyze if analysis $\geq \text{EQL}$
Reagent Blank	1 per Analytical Batch (Every 20 Samples)	$< L1 \text{ EQL}$	Reanalyze if analysis $\geq \text{EQL}$
Holding Time		60 days	

Representativeness and Comparability

Representativeness is defined as the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, process condition, or an environmental condition. Comparability is defined as expressing the confidence with which one data set can be compared to another.

It is recognized that the usefulness of the data is also contingent upon meeting the criteria for representativeness and comparability. Representativeness will be ensured by consistent use of standard sample collection, sample storage, sample packaging, sample transport, and laboratory sub-sampling procedures. Comparability will be ensured by using standard analytical methods and procedures, and QC and sample duplicates.

4.2.4 Data Review and Verification Requirements

Data verification is the process of accepting or rejecting data on the basis of established criteria. The QC personnel will use verification methods and criteria appropriate to the type of data, even those judged to be an "outlying" or spurious value. The persons verifying the data will have sufficient knowledge of the sampling and analytical methods to identify questionable values and deviations from criteria specified in relevant SOPs, Battelle Test Plan, CEMO Quality Manual or the TOCDF LQCP.

QC personnel, using criteria outlined in this document and applicable SOPs, will verify analytical and sampling data. The results from the laboratory QC samples will be used to further verify analytical results. QC personnel will perform review of items from the Sample Analysis Management Form, associated sampling records, analytical instrument raw data, Chains of Custody (COC), and analytical reports to verify completeness and accuracy of the data. Calculated results will be provided by software that is validated and controlled.

The following criteria will be used to evaluate the field sampling data:

- Use of approved sampling procedures
- Proper sampling per the SOP
- Use of properly operating and calibrated equipment
- Proper sample traceability maintained

The criteria listed below will be used to evaluate analytical data:

- Use of approved analytical procedures
- Use of properly operating and calibrated instrumentation

- Precision and accuracy achieved should be comparable to that achieved in previous analytical programs and consistent with the objectives stated in the Battelle Test Plan or TOCDF LQCP

4 2 5 Documentation and Records

Analytical results for individual samples will be generated and reported IAW the respective analytical methods, and will be filed by EG&G for future reference

Personnel will use standardized forms and laboratory record books to ensure completeness, traceability, and comparability of the process information and samples collected. A second person will conduct field checks of the standardized forms and records to ensure accuracy and completeness. Verification will be documented.

4 2 6 Reports To Management

If any corrective action is required during the program, these actions will be reported immediately to the TOCDF/EG&G Area 10 Sampling Manager. If the TOCDF/EG&G Area 10 Sampling Manager determines that a sampling event should be repeated, the decision will be made at that point and will be communicated to those involved.

4 3 Deliverables

4 3 1 Data Packages

Analytical reports, field records and supporting documentation will be produced by several organizations including DCD, CARA West, Battelle, and EG&G. These reports will be submitted to the EG&G PM for final approval. Raw data (e.g., mass spectra, chromatograms, calibrations, electronic files, etc.) will be maintained by the testing facility and will be made available upon request. The final completed record package (to include raw data as needed to support conclusions) will be stored by EG&G in accordance with PRP DC-004, Receipt and Storage of Records and Reference Documents. Data reported from the HMRC will include a case narrative section, Analytical Data Summary Sheets, QC Sample Results, COC forms, and copies of SOPs. Raw data will be maintained at the HMRC if any questions on the raw data arise.

4 3 2 Analytical Data Format

Data for analytes that are not detected will use the EQL for the lower reporting limit, and will be reported using "less than" values. The EQL will be defined as the quantitation level that corresponds to the lowest level at which the entire analytical system gives reliable signals or an acceptable calibration point or low-level matrix spike. Each compound or element is assigned a EQL that is contingent upon the behavior of the compound or element during analysis. Changes to extraction protocol, amount of sample prepared, or dilution applied to the sample can raise or lower the EQL.

4 3 3 Final Report

A complete Final Report describing the goals, methods, and results for the sampling project will be prepared and submitted to DSHW. Any deviation from this sampling plan will be documented and the potential impact to quality must be explained. Included in the report will be the following sections:

- Executive Summary – A concise overview of the project with a summary of the DQO results
- Introduction – A discussion of the project background and objectives
- Summary and Discussion of Results – A presentation of all pertinent project results including operations data, sampling results and summaries
- Sampling Methods – A brief description of the sampling methods used during the sampling project
- Analytical Methods – A brief description of the procedures used in analyzing the agent samples
- Quality Assurance/Quality Control – A summary of the pertinent QA/QC results relating to the analysis of the project's blanks, samples, duplicates, and matrix spike recoveries
- Appendices – A presentation of all remaining project-related data including all field sampling and recovery data sheets, laboratory analytical reports, sample tracking forms, calibration data, and SOPs
- Additionally, a QA summary will be supplied with the report to address all QA activities on the project, including
 - Corrective actions,
 - Data evaluations, and
 - Deviations from proposed protocols with a rationale for these deviations

4 3 4 Usage of Final Report

The final report will be used to satisfy the following objectives:

- Purity and HRA metals data will be used for agent characterization to eliminate the sampling prior to feeding during processing
- Headspace and liquid results will be used to determine the disposal pathway for “transparency” TCs. If Lewisite is detected, the “transparency” TC will be processed through the “drain and rinse” process associated with the Area 10 liquid incinerator

4 4 References

- 1 CARA West SOP TU-0000-M-076, Mobile Glove Box Systems
- 2 TOCDF LOP TE-LOP-557, Analysis of Metals by ICP-MS
- 3 SW-846 Method 9045D, Soil and Waste pH
- 4 HMRC IV-055-05, Purity and Impurity Analysis of Solutions Containing Chemical Agent by Gas Chromatography
- 5 HMRC IV-056-11, Operation and Maintenance of Gas Chromatography and Analysis of Solutions Containing GA, GB, GD, GF, HD, L, and VX by Gas Chromatography
- 6 HMRC IV-067-04, Quantitative Analysis of HD, GA, GB, GD, and VX Collected on Solid Sorbent
- 7 ECBC-TR-531 Appendix - Standard Operating Procedure for "Multi-Residue Quantitative Analysis of HD, HN3, Lewisite and Other Arsenical Chemical Warfare Agents in Permanganate-Based Demilitarization Waste Streams"
- 8 Battelle Chemical Environmental and Materials Operations (CEMO) Quality Manual

Appendix C

Test Plan and Test Plan Change Memos

Test Plan
For
Ton Container Sample Analysis

June 17, 2009

Prepared By
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Ton Container Sample Analysis

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Ton Container Sample Analysis

Ton Container Sample Analysis

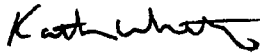
Approvals



Thomas A. Malloy IV
Battelle Senior Research Scientist

06/17/09

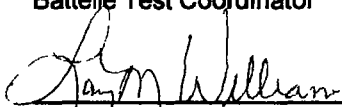
Date



Katherine Whittington
Battelle Test Coordinator

06/17/09

Date



Larry Williams
EG&G Project Manager

6/18/09

Date

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ACRONYMS

GC-MSD	gas chromatograph – mass selective detector
MDL	method detection limit
MS	mass spectrometer
ACL	analytical chemistry laboratory
AMC	Army Materiel Command
BCO	Battelle Columbus Operations
BFG	4-bromofluorobenzene
CA	chemical agent
CCV	continuing calibration verification
CEMO	Chemical, Environmental and Materials Operations
CoC	chain of custody
CSM	chemical surety material
CW	chemical warfare
DFTPP	decafluorotriphenylphosphine
ECBC	Edgewood Chemical and Biological Center
EQL	estimated quantitation limit
GA	tabun
GC	gas chromatography (chromatograph)
GC-FPD	gas chromatograph – flame photometric detector
GC-MS	gas chromatograph – mass spectrometer
HAZOP	hazardous operations
HML	hazardous materials laboratory
HMRC	Battelle's Hazardous Materials Research Center
HRA	Health Risk Assessment
HTM	highly toxic material
L	Lewisite
L-1	2-Chlorovinylarsonous dichloride
L-2	Bis(2-chlorovinyl)arsinous chloride
L-3	Tris(2-chlorovinyl)arsine
LITF	large item test facility
LRB	laboratory record book
MS	mass spectrometer
OSHA	Occupational Safety and Health Administration
PI	Principal Investigator
PFTBA	perfluorotributylamine
PM	Program Manager
PPE	personal protective equipment
PQL	practical quantitation limit
QA	Quality Assurance
RDTE	research, development, testing and evaluation
SOP	standard operating procedure
TIC	tentatively identified compounds
TMP	2,2,4-trimethylpentane
TOCDF	Toole Chemical Agent Destruction Facility
TPCS	test performance control sheet
VOC	volatile organic compound

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1 0 INTRODUCTION

1 1 Background

Deseret Chemical Depot currently stores ten ton containers (TCs) of Lewisite (L) comprising approximately 13 tons of agent and four ton containers of GA. Additionally there exists ten TCs of what is described as "transparency" TCs. The latter are believed to have contained Lewisite at one time, and have since been decontaminated and are considered empty. There are conflicting data, both documented and anecdotal, concerning the actual contents of these TCs. EG&G, DMI has been tasked by the Chemical Materials Agency (CMA) to develop means to destroy the agent and decontaminate the drained and transparency TCs for final disposal. EG&G has proposed incinerating the Lewisite from the TCs. The existing baseline incinerator systems would be unable to destroy the Lewisite in a timely and cost effective method, therefore EG&G has proposed modifying an existing small incinerator system built to destroy stocks of sulfur mustard. The incinerator system will be modified to remove the arsenic, mercury and other metals that are prevalent in either the chemical structure of Lewisite or as a contaminate from the previous fill of the TCs.

The GA/Lewisite Sampling Program will collect representative samples from the GA, GA/UCON, Lewisite and "transparency" TCs to support the processing in a liquid incinerator system to be constructed in Area 10 of the DCD. These samples will be supplied to Battelle's Hazardous Materials Research Center (HMRC) for preparation and analysis. The results of the analysis will allow engineering plans and controls to be added to the planned disposal system to aid the incineration of the agent and the clearing of the TCs for off-site disposal.

The drained Lewisite TC will also need to be treated in order to meet the conditions of the Organization for the Prevention of Chemical Weapons (OPCW) treaty and also to meet the U.S. Army's conditions for off-site commercial disposal. EG&G plans to meet these treatment conditions using a series of rinses. The drained Lewisite TCs will be rinsed with 20% acetic acid solution that will act as an organic solvent and a carrier for the remaining Lewisite to be destroyed in the incinerator. One or more rinses with the acetic acid may be necessary. Following the acetic acid rinse(s) the TC will be rinsed one or more times with a 7.0 M nitric acid solution. The nitric acid will dissolve any remaining metals in the liquid, oxidize any Lewisite and remove the embedded metals in the pores of the TC.

1 2 Objectives

To sufficiently characterize GA and Lewisite agent feed to the liquid incinerator in a manner that is acceptable to the State of Utah, Division of Solid and Hazardous Waste Characterization tests will include

- agent purity,
- tentatively identified compounds (TIC),
- density,
- pH,
- chlorobenzene content,

Ton Container Sample Analysis

- L1, L2 and L3 content,
- Health Risk Assessment (HRA) metals and
- volatile organic compounds (VOC)

The objective of the rinse testing is to be able to use the test results to support the proposed rinsing of the ton containers

1 3 Limitations

Because stock Lewisite, containing primarily L1, will be used to prepare instrument calibration standards only L1 will be quantitatively determined in samples. The presence of L2 and L3 will be qualitatively determined.

Only a single sorbent tube headspace sample will be collected from each "transparency" ton container, therefore, reanalysis of these samples will not be possible as the entire sample is consumed during the thermal desorption process.

1 4 Expected Use of Results

For the characterization tests the expected use of results is the collection of sufficient data for the processing of ton containers without the need to collect additional samples prior to agent destruction.

For the ton container rinse test the expected use of results is to

- Verify that analytical methods can support the proposed rinsing of the TC
- Verify the end state of the metal of the TC is sufficiently clean to support the OPCW treaty requirement and for off-site disposal
- Support the process design

2 0 ORGANIZATION

Battelle's Hazardous Materials Research Center (HMRC), located in West Jefferson, Ohio, is part of Chemical, Environmental and Materials Operations (CEMO). The HMRC is an ISO 9001-certified facility that provides a broad range of materials testing, system and component evaluation, research and development, and analytical chemistry services requiring the safe usage and storage of highly toxic substances. Since its initial certification by the U.S. Army in 1981, the facility has functioned as both a research and a technology development laboratory in support of DoD chemical defense programs. The HMRC can safely store and handle all traditional chemical warfare (CW) agents, other highly toxic materials (HTMs), agent simulants, Class A poisons, and toxins. All work is managed under a current U.S. Army Bailment Agreement and undergoes periodic inspections by the Edgewood Chemical and Biological Center (ECBC), the Army Materiel Command (AMC), and the Department of the Army Inspector General.

2 1 Personnel

Tom Malloy – HMRC Program Manager

The responsibilities of the program manager include

- Coordinate testing
- Maintain communication with the client (internal and/or external)
- Prepare the draft test plan and reports
- Direct the effort to ensure that budget and schedule are met
- Coordinate for use of the test facility and establish a test schedule
- Coordinate for the availability of qualified staff to conduct the tests
- Revise the test plans and reports in response to reviewers' comments
- Coordinate distribution of final test plans and reports

Gary Stickel – Peer Reviewer

The responsibilities of the peer reviewer include

- Review and/or aid in the preparation the draft test plan and reports
- Act as a subject matter expert on technical issues

Elizabeth Balaban – HMRC Principal Investigator

The responsibilities of the principal investigator include

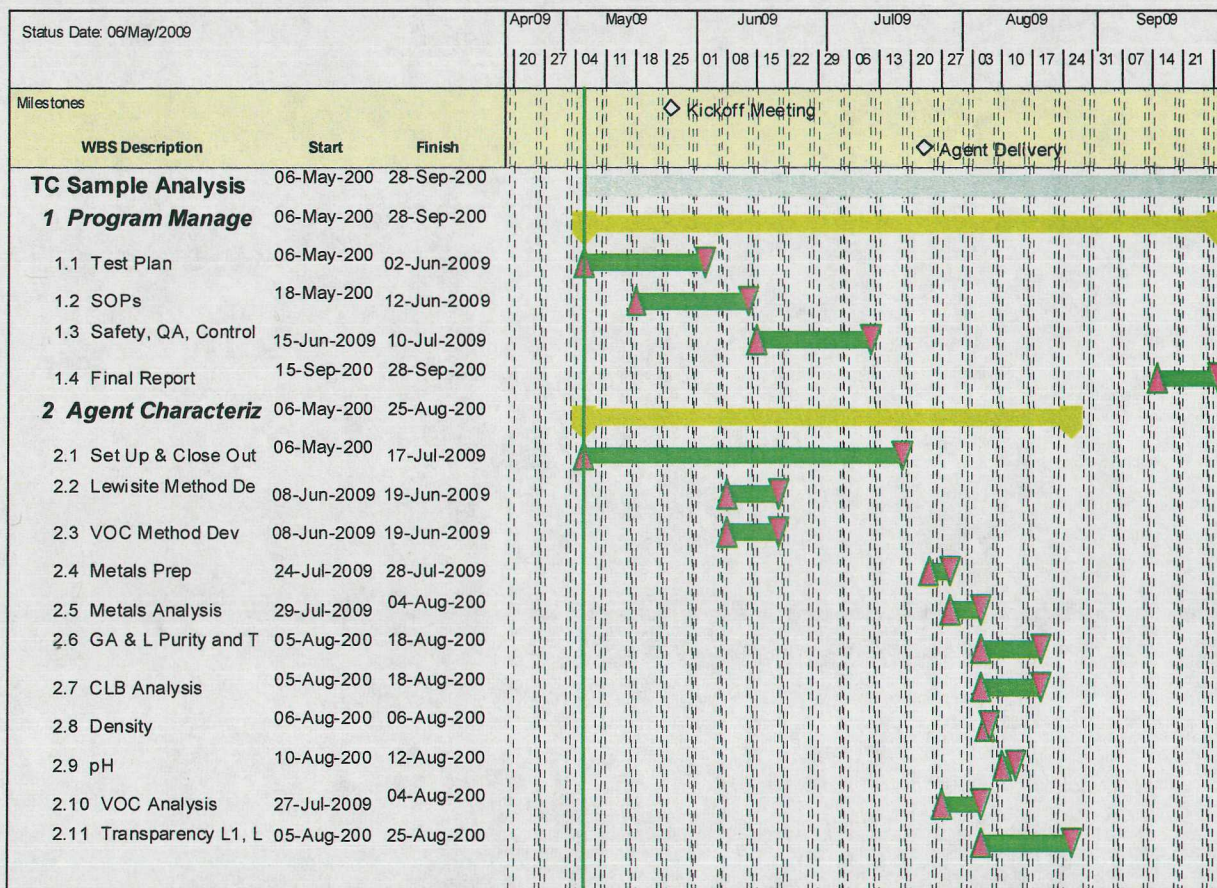
- Have overall responsibility for ensuring that the test plan is followed
- Keep the HMRC Program Manager informed of progress and difficulties in conducting the tests
- Respond to any issues raised in reports, including instituting corrective action as necessary
- Direct the effort to ensure that budget and schedule are met
- Conduct a technical review of the draft test plan and reports

2.2 Facilities

Sample receipt and preparation will be conducted at Battelle HMRC, a complex that consists of approximately 30,000 sq. ft., which includes the Hazardous Materials Laboratory (HML), the Large Item Test Facility (LITF), and the Analytical Chemistry Laboratory (ACL). The HML consists of eight laboratory areas that provide approximately 3,500 sq. ft. of laboratory space and 23 chemical surety material (CSM)-approved filtered hoods (156 linear ft.) for working with neat (pure) CSM. The LITF consists of approximately 500 sq. ft. of laboratory space for testing items and systems too large to fit into standard laboratory fume hoods. The ACL consists of 2,000 sq. ft. of laboratory space, 12 gas chromatographs (GC) with both flame photometric detectors (FPD) and flame ionization detectors (FID), and 4 GCs with mass selective detectors (MSD) for the analytical characterization of samples in support of all activities conducted in the HML and the LITF. The ACL has four fume hoods (22 linear ft.) for preparing analytical standards and analyzing test samples that meet the defined concentration levels for dilute solutions (i.e., diluted chemical agent).

2.3 Schedule

Sample collection will be performed by CARA West starting on July 7, 2009. Delivery of samples is scheduled for July 23, 2009. The sample testing and delivery of the final report, show in the below schedule, is predicated upon this delivery date.



Status Date: 06/May/2009			Apr09		May09				Jun09				Jul09				Aug09				Sep09				
WBS Description	Start	Finish	20	27	04	11	18	25	01	08	15	22	29	06	13	20	27	03	10	17	24	31	07	14	21
3 TC Rinse Test	17-Aug-200	14-Sep-200																							
3.1 Set Up and Solubilit	17-Aug-200	19-Aug-200																							
3.2 Test Execution	24-Aug-200	27-Aug-200																							
3.3 Lewisite Prep & Ana	31-Aug-200	04-Sep-200																							
3.4 Metals Prep & Analy	31-Aug-200	14-Sep-200																							
4 Calorimetry	17-Aug-200	28-Aug-200																							
4.1 Set Up	17-Aug-200	21-Aug-200																							
4.2 Training & Test Exe	24-Aug-200	28-Aug-200																							
5 Materials	25-May-200	12-Jun-2009																							

2.3.1 Intermediate Deliverables / Milestones

- Draft test plan
- Final test plan
- Final report

3.0 APPROACH

3.1 Overview

The first part of this testing will involve characterizing liquid and sludge samples collected from 4 GA ton containers and 10 Lewisite ton containers. Liquid and air samples collected from 10 "transparency" ton containers, believed to be free of chemical agent, will also be analyzed. The second part of this testing will involve evaluating a procedure designed to mimic the proposed rinsing of residual Lewisite from drained ton containers. This testing will also involve collecting calorimetric data collected from the mixing Lewisite with acetic acid and an acetic-Lewisite mixture with nitric acid.

3.2 Equipment and Materials

The following equipment and materials will be provided by Battelle for use in testing.

- CEM Mars eXpress microwave
- Microwave vessels
- DAAMS tubes
- Ball mills
- 2 L polycarbonate, wide-mouth bottles
- Mettler RC1e reaction calorimeter
- 80 mL reaction vessel
- Mass flow meter
- MiniWarn detector

Agent for the preparation of calibration and spiking standards will be provided by TOCDF through ECBC or transferred to this task from other TOCDF projects

- GA (CAS # 77-81-6) with a purity >85%
- Lewisite [primarily L-1 (CAS # 541-25-3) with mixture of L-2 (40334-69-8) and L-3 (40334-70-1)] with a combined purity >85%
- Chlorobenzene (CAS # 108-90-7) with a purity >99%
- 2,2,4-trimethylpentane (CAS # 540-84-1)
- Ethanethiol (CAS # 75-08-1)
- Decafluorotriphenylphosphine (DFTPP) (CAS # 5074-71-5)
- 4-Bromofluorobenzene (BFB) (CAS # 460-00-4)
- Nitric acid – trace metals grade
- Hydrochloric acid – trace metals grade
- Acetic acid – trace metals grade
- Reagent water – trace metals grade

3 3 Agent Receipt and Accountability

Receipt and accountability of agent samples will adhere to SOP HMRC I-023-14 "Chemical Agent (CA) Receipt, Storage, Accountability and Reporting at the HMRC"

All agent samples will be decontaminated and disposed of following acceptance of the final report

3 4 Test Matrix

Eight different tests will be performed to characterize agent samples, as shown in Table 1. The following types and numbers of samples will be received:

- GA liquid – 4 samples plus 1 field duplicate
- GA sludge – 0 to 4 samples
- Lewisite liquid – 10 samples plus 1 field duplicate
- Lewisite sludge – 0 to 10 samples
- Transparency ton liquid – 0 to 10 samples plus 0 to 1 field duplicate
- Transparency ton vapor – 0 to 10 samples

Not all samples will be analyzed by all tests. See section 3.5 for a description of tests to be performed and Appendix A for a table of samples to be collected and tests to be performed.

Table 1 GA and L Characterization Sample Matrix

Prep & Analysis	Method	GA Liquid	GA Sludge*	L Liquid	L Sludge*	Transparency Ton Liquid*	Transparency Ton Vapor
pH	9045D	4	-	10	-	-	-
Density	Volume and Mass	4	-	10	-	-	-
Purity	HMRC IV-055	4	-	10	-	-	-
TICs	HMRC IV-055	4	-	10	-	-	-
Chlorobenzene	HMRC IV-056	4	-	-	-	-	-
L1, L2 and L3	ECBC-523	-	-	-	-	10	-
VOCs	HMRC IV-067	-	-	-	-	-	10
HRA Metals	Project Specific SOP	4	4	10	10	10	-

* maximum number of samples – may be fewer

Table 2 shows the samples that will be collected for analysis during the ton container rinse test. Samples will be analyzed for each lewisite homologue L1, L2 and L3, as well as arsenic (As) and mercury (Hg). Two tests (A and B) will be performed in parallel. See Section 3.6 for a description of the tests to be performed.

Table 2 Lewisite Ton Container Rinse Test Sample Matrix

Sample Name	Container No	Step (App A)	L1, L2, L3	As & Hg
HOAC-1 A/B	1 A/B	8	1	-
Coupon 1 A/B	1 A/B	8	1	1
HOAC-1 A/B	1 A/B	9	1	-
HOAC-1 A/B	1 A/B	10	1	-
HOAC-2 A/B	2 A/B	17	1	-
HOAC-1/HNO3-1 A/B	3 A/B	17	1	-
HOAC-2 A/B	2 A/B	21	1	-
HOAC-1/HNO3-1 A/B	3 A/B	21	1	-
HOAC-2 A/B	2 A/B	22	1	-
HOAC-1/HNO3-1 A/B	3 A/B	22	1	-
HOAC-3 A/B	4 A/B	29	1	-
HOAC-2/HNO3-2 A/B	5 A/B	29	1	-
HOAC-3 A/B	4 A/B	32	1	-
HOAC-2/HNO3-2 A/B	5 A/B	32	1	-
HOAC-3 A/B	4 A/B	33	1	-
HOAC-2/HNO3-2 A/B	5 A/B	33	1	-
HOAC-3/HNO3-3 A/B	6 A/B	38	1	1
Coupon 2 A/B	6 A/B	38	1	1
HOAC-3/HNO3-3 A/B	6 A/B	40	1	1
HOAC-3/HNO3-3 A/B	6 A/B	41	1	1

Sample Name	Container No	Step (App A)	L1, L2, L3	As & Hg
HOAC-3/HNO3-4 A/B	7 A/B	46	1	1
Coupon 3 A/B	7 A/B	46	1	1
HOAC-3/HNO3-4 A/B	7 A/B	48	1	1
HOAC-3/HNO3-4 A/B	7 A/B	49	1	1
HOAC-3/HNO3-5 A/B	8 A/B	54	1	1
Coupon 4 A/B	8 A/B	54	1	1
HOAC-3/HNO3-5 A/B	8 A/B	56	1	1
HOAC-3/HNO3-5 A/B	8 A/B	57	1	1
TR-1 A/B	9 A/B	61	1	1
TR-2 A/B	10 A/B	67	1	1
TR-3 A/B	11 A/B	73	1	1
Total Samples			31	16

3 5 Procedures - GA and L Characterization

3 5 1 pH for GA, Lewisite liquid, and "transparency" TC liquid samples

pH will be determined following EPA Method 9045D using a pH meter. A 1.0 g sample will be used instead of the specified 20 g sample. There is no estimated quantitation limit (EQL) for this method. See Table 3 for method QA/QC criteria. See Appendix A for identification of samples for analysis.

Table 3 QA/QC Criteria for Determination of pH for GA and L

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Initial Calibration	Daily prior to use		
Continuing Cal Checks	Every 2 hr		
Internal Standards	None		
MS/MSD	None		
Sample Duplicate	1 per sample	RPD \leq 15%	Check Calculations, Assess Impact on Data, Narrate
Blank Spike	None		
Method Blank	None		
Reagent Blank	1 per sample		
Holding Time		60 days	

3 5 2 Density for GA and Lewisite liquid samples

A Class A syringe will be used to deposit a known volume of agent onto a balance capable of measuring to 0.1 mg. Balance will be calibrated with NIST traceable weights. Measurement will be performed at ambient temperature. Density will be determined as measured mass divided by measured volume. Density will not be performed on Lewisite or GA sludge samples. There

is no EQL for this method See Table 4 for method QA/QC criteria See Appendix A for identification of samples for analysis

Table 4 QA/QC Criteria for Determination of Density for GA and L

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Initial Calibration	Daily prior to use (balance)		
Continuing Cal Checks	None		
Internal Standards	None		
MS/MSD	None		
Sample Duplicate	1 per sample	RPD \leq 10%	Check Calculations, Assess Impact on Data, Narrate
Blank Spike	None		
Method Blank	1 per Analytical Batch (Every 20 Samples - using deionized water)		
Reagent Blank	None		
Holding Time		60 days	

3 5 3 Agent purity for GA liquid samples

Purity will be determined following SOP HMRC IV-055 "Purity and Impurity Analysis of Solutions Containing Chemical Agent by Gas Chromatography" Purity will be reported as a percentage on a mass basis There is no EQL for this method See Table 5 for method QA/QC criteria See Appendix A for identification of samples for analysis

Table 5 QA/QC Criteria for Determination of GA purity by GC/FID

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Initial Calibration	None		
Continuing Cal Checks	None		
Internal Standards	None		
MS/MSD	None		
Sample Duplicate	1 per Analytical Batch (Every 20 Samples)	RPD \leq 25%	Check Calculations, Assess Impact on Data, Narrate
Blank Spike	None		
Method Blank	None		
Reagent Blank	1 per sample	Used to baseline correct sample response	
Holding Time		60 days	

3 5 4 TICs for GA liquid samples

Tentatively identified compounds will be determined following SOP HMRC IV-055 Samples will be analyzed by full scan GC/MS A NIST 2002 mass spectral library will be used to evaluate samples for TICs Reports will be generated using ChemStation software There is no EQL for

this method See Table 6 for method QA/QC criteria See Appendix A for identification of samples for analysis

Table 6 QA/QC Criteria for Determination of TICs in GA and L Liquids by GC/MS

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Instrument Tune (PFTBA)	Initially and as required	Method Key Ion and Ion Abundance Criteria	Repeat Instrument Tune
Instrument Evaluation and Performance Check (50 µg/ml DFTPP)	Initially, Daily, and as Required	EPA Method 8270C DFTPP Criteria	Evaluate System, Perform Corrective Maintenance Recalibrate
Initial Calibration	None		
Continuing Calibration Checks	None		
Internal Standards	None		
MS/MSD	None		
Sample Duplicate	1 per Analytical Batch (Every 20 Samples)	Qualitative comparison between samples	
Blank Spike	None		
Method Blank	None		
Reagent Blank	1 per sample	Baseline comparison to sample data	
Holding Time		60 days	

3 5 5 Chlorobenzene for GA liquid samples

GA liquid samples will be analyzed by GC/MS following guidelines of SOP HMRC-056 "Operation and Maintenance of Gas Chromatography and Analysis of Solutions Containing GA, GB, GD, GF, HD, L, and VX by Gas Chromatography" The EQL for this method is 1 mg/mL based on 10 µL sample diluted in 10 mL of solvent with a low calibration point of 1 0 µg/mL See Table 7 for method QA/QC criteria See Appendix A for identification of samples for analysis

Table 7 QA/QC Criteria for Determination of Chlorobenzene in GA by GC/MS

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Instrument Tune (PFTBA)	Initially and as required	Method Key Ion and Ion Abundance Criteria	Repeat Instrument Tune
Instrument Evaluation and Performance Check (50 µg/ml DFTPP)	Initially, Daily, and as Required	EPA Method 8270C DFTPP Criteria	Evaluate System, Perform Corrective Maintenance Recalibrate
Initial Calibration	5 point calibration of CLB	$R^2 \geq 0.990$ Each Level $\pm 15\%$ of Theoretical Value	Evaluate System Recalibrate System
Continuing Calibration Checks	Low calibration standard	Within $\pm 25\%$	Reanalyze If outside $\pm 25\%$, Perform

Quality Parameter	Method/Frequency	Criteria	Corrective Action
	concentration and second-highest calibration standard concentration, independently prepared, Every 5 Samples		maintenance on Instrument Recalibrate Reanalyze Affected Samples
Internal Standard	Each sample	Within ± 30 Seconds of Continuing RT Value	Check Sensitivity of System Reanalyze samples or standards
MS/MSD	1 per Analytical Batch (Every 20 Samples)	Recovery 50 to 150% RPD $\leq 25\%$	Check Calculations, Assess Impact on Data, Narrate
Sample Duplicate	1 per Analytical Batch (Every 20 Samples)	RPD $\leq 25\%$	Check Calculations, Assess Impact on Data, Narrate
Blank Spike	1 per Analytical Batch (Every 20 Samples)	Recovery 50 to 150%	Check Calculations, Assess Impact on Data, Narrate
Method Blank	1 per Analytical Batch (Every 20 Samples)	< EQL (PQL)	Reanalyze if analysis \geq EQL (PQL)
Reagent Blank	1 per Analytical Batch (Every 20 Samples)	< EQL (PQL)	Reanalyze if analysis \geq EQL (PQL)
Holding Time		60 days	

3 5 6 Agent purity and TICs for Lewisite liquid samples

Samples will be dissolved in acetone and derivatized as described in Section 3 5 7 Analysis will be performed by GC/MS per SOP HMRC IV-055 to determine the retention time (RT) of Lewisite 1 and 2, identified as their thioether derivatives, and Lewisite 3 A NIST 2002 mass spectral library will be used to evaluate samples for TICs Reports will be generated using ChemStation software GC/MS RT data will be applied to purity analysis by GC/FID per SOP HMRC IV-055 Purity will be reported as a percentage on a mass basis There is no EQL for this method See Table 8 and Table 6 for method QA/QC criteria See Appendix A for identification of samples for analysis

Table 8 QA/QC Criteria for Determination of L purity by GC/MS and GC/FID

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Instrument Tune (PFTBA)	Initially and as required	Method Key Ion and Ion Abundance Criteria	Repeat Instrument Tune,
Instrument Evaluation and Performance Check (50 μ g/ml DFTPP)	Initially, Daily, and as Required	EPA Method 8270C DFTPP Criteria	Evaluate System, Perform Corrective Maintenance Recalibrate
Initial Calibration	None		
Continuing Calibration Checks	None		

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Internal Standards	None		
MS/MSD	None		
Sample Duplicate	1 per Analytical Batch (Every 20 Samples)	RPD \leq 25%	Check Calculations, Assess Impact on Data, Narrate
Blank Spike	None		
Method Blank	None		
Reagent Blank	1 per sample	Used to baseline correct GC/FID sample response	
Holding Time		60 days	

3 5 7 L1, L2 and L3 for “transparency” ton liquid samples

Samples will be prepared and analyzed in accordance with the procedures reported in ECBC-TR-531 for the analysis of L1, L2 and L3 GC/MS calibration standards will be prepared from Lewisite stock available at the HMRC. As this Lewisite stock contains primarily L1, quantitative analysis of rinse samples for L2 and L3 utilizing methodology described in ECBC-TR-531 may not be possible, semi-quantitative analysis may be feasible.

The MDL reported for L1 by this method is 25 µg/L. An MDL study for L1 will be performed per 40 CFR Part 136 Appendix B. The estimated quantitation limit for a 0.5 mL sample extracted into 5.0 mL of 2,2,4-trimethylpentane (TMP) is 150 µg/L. Direct quantification of L2 and L3 may not be possible, however these compounds will be identified based on the spectral identification of their components. See Table 9 for method QA/QC criteria. See Appendix A for identification of samples for analysis.

Table 9 QA/QC Criteria for Determination of L1, L2 and L3 in Transparency Ton Liquid Samples by GC/MS

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Instrument Tune (PFTBA)	Initially and as required	Method Key Ion and Ion Abundance Criteria	Repeat Instrument Tune
Instrument Evaluation and Performance Check (50 µg/ml DFTPP)	Initially, Daily, and as Required	EPA Method 8270C DFTPP Criteria	Evaluate System, Perform Corrective Maintenance, Recalibrate
Initial Calibration	5 point calibration of L1	$R^2 \geq 0.990$ Each Level $\pm 15\%$ of Theoretical Value	Evaluate System, Recalibrate System
Continuing Calibration Checks	Low calibration standard concentration and second-highest calibration standard concentration, independently prepared, Every 5	Within $\pm 25\%$	Reanalyze. If outside $\pm 25\%$, Perform maintenance on Instrument, Recalibrate, Reanalyze Affected Samples

Quality Parameter	Method/Frequency	Criteria	Corrective Action
	Samples		
Internal Standards	None		
MS/MSD	1 per Analytical Batch (Every 20 Samples)	Recovery 50 to 150% RPD \leq 25%	Check Calculations, Assess Impact on Data, Narrate
Sample Duplicate	1 per Analytical Batch (Every 20 Samples)	RPD \leq 25%	Check Calculations, Assess Impact on Data, Narrate
Blank Spike	1 per Analytical Batch (Every 20 Samples)	Recovery 50 to 150%	Check Calculations, Assess Impact on Data, Narrate
Method Blank	1 per Analytical Batch (Every 20 Samples)	< L1 EQL	Reanalyze if analysis \geq EQL
Reagent Blank	1 per Analytical Batch (Every 20 Samples)	< L1 EQL	Reanalyze if analysis \geq EQL
Holding Time		60 days	

3 5 8 Headspace VOCs for “transparency” ton sorbent tube samples

Solid sorbent tubes samples collected from ton container headspace for qualitative VOC identification and semi-quantitative analysis will be thermally desorbed and analyzed by GC/MS following guidelines of SOP HMRC-067 “Quantitative Analysis of HD, GA, GB, GD, and VX Collected on Solid Sorbent” The suitability of this procedure for the analysis of VOCs will be demonstrated by the analysis of a commercially available mixture containing 53 VOCs concurrent with sample analysis Components in both samples and the commercial VOC mix will be qualitatively identified by mass spectral library match using Agilent ChemStation software designed for the analysis of TICs Semi-quantitative results of the VOC TICs will be reported using ChemStation by comparison to a deuterated internal standard There is no EQL for this method See Table 10 for method QA/QC criteria See Appendix A for identification of samples for analysis

Table 10 QA/QC Criteria for Determination of VOCs in TC Headspace by GC/MS

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Instrument Tune (PFTBA)	Initially and as required	Method Key Ion and Ion Abundance Criteria	Repeat Instrument Tune
Instrument Evaluation and Performance Check (50 µg/ml BFB)	Initially, Daily, and as Required	EPA Method 8260C BFB Criteria	Evaluate System, Perform Corrective Maintenance Recalibrate
Initial Calibration	None		
Continuing Calibration Checks	None		
Internal Standards	Each Sample	Within \pm 30 Seconds of Continuing RT Value	Check Sensitivity of System
MS/MSD	None		

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Sample Duplicate	None		
Blank Spike	None		
Method Blank	1 per Analytical Batch (Every 20 Samples)	Baseline comparison to sample data	
Reagent Blank	None		
Holding Time		28 days	

3 5 9 HRA Metals for GA liquid, GA sludge, Lewisite liquid, Lewisite sludge, and “transparency” ton liquid samples

Samples will be prepared (digested) at the HMRC and analyzed at Battelle’s laboratory at King Ave , Columbus, OH Sample preparation and analysis will follow a project specific SOP based on TE-LOP-557 and TE-LOP-584 The EQLs for the target metal analytes are summarized in Table 11 EQLs are based on the digestion of 0.2 g of sample with a final sample volume of 100 mL prior to analysis Samples will be below research, development, testing and evaluation (RDTE) dilute solution limits for GA and L based on starting agent mass and final sample volume Transparency ton liquid samples will be prepared in the same manner as the liquid agent and sludge samples and analyzed on a weight basis Actual EQLs for the samples will be dependent on the sample weight and preparation dilution factor See Table 12 for method QA/QC criteria See Appendix A for identification of samples for analysis

Table 11 Target HRA Metals and EQLs

HRA Metal	EQL (mg/kg)
Aluminum	5
Antimony	0.05
Arsenic	0.05
Barium	0.05
Beryllium	0.05
Boron	5
Cadmium	0.05
Chromium	0.05
Cobalt	0.05
Copper	0.05
Lead	0.05
Manganese	0.05
Mercury	0.25
Nickel	0.05
Selenium	1.25
Silver	0.5
Thallium	0.05
Tin	0.05
Vanadium	0.05
Zinc	5

Table 12 QA/QC Criteria for Determination of HRA Metals

Quality Parameter	Method/Frequency	Criteria¹
ICS	Beginning of analytical run or once every 12 hours, whichever is more frequent	70% to 130% recovery
CCB	At least once every 10 analytical samples	Response < 3 x IDL
CCV	At least once every 10 analytical samples	Results \pm 10% of initial calibration
Preparation Blank	At least once every 20 analytical samples	< EQL
Dilution Test	At least once every 20 analytical samples	Within \pm 10% of original determination where elements are found within the linear calibration range of the instrument for both the parent and the dilution
PDS	At least once every 20 analytical samples	75% to 125% recovery
LCS	At least once every 20 analytical samples	\pm 25% of spike amount
MS	At least once every 20 analytical samples	\pm 25% recovery
MSD	At least once every 20 analytical samples	\leq 20% RPD
Sample Duplicate	At least once every 20 analytical samples	\leq 20% RPD if response >100 IDL
Field Duplicate	One per TC type, e.g., (1) GA, GA/UCON, (2) Lewisite, and (3) "transparency", for liquid samples only	\leq 25% RPD if response >100 IDL
Holding Time	Every sample	28 Days (Mercury) 6 Months (All other metals)

¹ Corrective actions will be captured in the project specific SOP

3 6 Procedure – Lewisite Ton Container Rinse Test

3 6 1 Solubility Test

A preliminary solubility test will be performed by placing 1 0 mL of Lewisite in a clear container followed by 135 mL of 20% (w/v) acetic acid (HOAC) solution. The mixture will be placed on a ball mill and rolled at ambient temperature for at least 6 hr. Periodic observations will be made and recorded to evaluate whether the lewisite goes into solution or remains as a separate phase.

The sample will then be allowed to stand over night and observed for precipitation, settling, emulsion formation or phase separation. Photographs will be taken to record any physical changes.

3 6 2 Rinse Test

The stepwise procedure for the rinse test is presented in Appendix B. This procedure is identical in content to the one supplied in the Statement of Work, however, some step numbers have been changed to letters for ease of reading, no steps have been removed. This procedure will be performed in duplicate, however, addition of 20% acetic acid to the test containers will be accomplished by two means.

- Rinse Test A – add 1350 mL of 20% acetic acid
- Rinse Test B – add 810 mL of water and then add 540 mL of 50% acetic acid

Sample containers and names will be designated with an A or B for tracking. The rinse test will take three days to complete. Aqueous samples that need to be used on subsequent days of testing will be stored at 4±3°C overnight. Coupons will be either locked in the fume hood at room temperature or stored at 4±3°C overnight. Archived samples will be stored at room temperature and then disposed of following acceptance of the final report.

3 6 2 1 Determination of Lewisite Homologues

The Lewisite homologues (L1, L2 and L3) will be determined using the method described in ECBC-TR-531. As stated in Section 3 5 7 of this test plan, due to the availability of Lewisite stock, quantitative analysis of L1 and qualitative analysis of L2 and L3 will be performed. Note that the method does not distinguish between the lewisite and its corresponding trivalent acid, the value reported for L1 is the sum of L1/CVAA and the value reported for L2 is the sum of L2/BCVAA. See Table 13 for method QA/QC criteria. Note that QC samples will be performed at a higher frequency than for the transparency TC liquid analysis. Also, a blank spike duplicate will be performed. De-ionized water will be used as the matrix for the blank and blank spikes. The recovery of matrix spikes from nitric acid rinse samples may be low due to Lewisite reaction with the acid, recoveries below 50% for these samples will be noted in the final report but will not result re-preparation of the samples in the associated batch. Samples will be extracted within 24 hr of preparation and will be stored at 4±3°C until extracted.

Table 13 QA/QC Criteria for Determination of L1, L2 and L3 Rinse Samples by GC/MS

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Instrument Tune (PFTBA)	Initially and as required	Method Key Ion and Ion Abundance Criteria	Repeat Instrument Tune
Instrument Evaluation and Performance Check (50 µg/ml DFTPP)	Initially, Daily, and as Required	EPA Method 8270C DFTPP Criteria	Evaluate System, Perform Corrective Maintenance, Recalibrate
Initial Calibration	5 point calibration of L1	$R^2 \geq 0.990$ Each Level $\pm 15\%$ of Theoretical Value	Evaluate System, Recalibrate System
Continuing Calibration	Low calibration	Within $\pm 25\%$	Reanalyze If outside

Quality Parameter	Method/Frequency	Criteria	Corrective Action
Checks	standard concentration and second-highest calibration standard concentration, independently prepared, Every 5 Samples		±25%, Perform maintenance on Instrument Recalibrate Reanalyze Affected Samples
Internal Standards	None		
MS/MSD	1 per Analytical Batch (Every 10 Samples)	Recovery 50 to 150% RPD ≤ 25%	Check Calculations, Assess Impact on Data, Narrate
Sample Duplicate	1 per Analytical Batch (Every 10 Samples)	RPD ≤ 25%	Check Calculations, Assess Impact on Data, Narrate
Blank Spike / Blank Spike Duplicate	1 per Analytical Batch (Every 10 Samples)	Recovery 50 to 150%	Check Calculations, Assess Impact on Data, Narrate
Method Blank	1 per Analytical Batch (Every 10 Samples)	< L1 EQL	Reanalyze if analysis ≥EQL
Reagent Blank	1 per Analytical Batch (Every 10 Samples)	< L1 EQL	Reanalyze if analysis ≥EQL
Holding Time		60 days	

The typical working calibration range is 5 µg/L to 10,000 µg/L. Initial samples will contain elevated levels of Lewisite (up to ~14,000 mg/L) requiring either a reduced sample volume or sample dilution prior to analysis. Samples will need to be diluted to below RDTE dilute solution levels prior to transport to the HMRC RDTE analytical laboratory. The EQL for L1 is 150 µg/L.

The metal coupons will be analyzed for residual Lewisite (L1, L2 and L3) using the method described in ECBC-TR-531. All sample coupons will be extracted in a single batch. Since there will be a limited number of coupons available, two coupon blanks and two spiked coupons will be extracted and analyzed per test. Coupons will be extracted 2,2,4-trimethylpentane containing 1% ethanethiol by volume. Sufficient TMP solution will be used to cover the coupons. A consistent volume of TMP solution will be used for all tests. Coupon extracts will be analyzed in the same manner as the liquid samples. Results for L1 will be reported as µg/coupon. L2 and L3 will be reported as present or absent.

3.6.2.2 Determination of Total Arsenic and Mercury

Total arsenic and mercury in the liquid samples will be determined by ICP-MS following the project specific SOP based on TE-LOP-557 and TE-LOP-584. The EQL for arsenic and for mercury is 0.1 µg/L based on a sample volume of 45 mL. See Table 12 for method QA/QC criteria.

Following extraction for residual Lewisite, recoverable arsenic and mercury on the TC coupons will be determined using the same ICP-MS method after extraction with 0.1 N HNO₃. Samples will be heated to ~50°C to enhance extraction and then analyzed. Results for As and Hg will be

reported as mg/coupon. All sample coupons will be extracted in a single batch along with two blank coupons and two spiked coupons.

3.6.3 Reaction Calorimetry Tests

Solution calorimetry will be performed to obtain heat of reaction/dissolution data and to calculate pressure data when Lewisite is mixed with a 20% (w/v) acetic acid solution and when a representative acetic acid/Lewisite solution is mixed with a 7.0 M nitric acid solution. The testing will be performed using a Mettler RC1e reaction calorimeter with an 80 mL reaction vessel. The test temperature will be $21 \pm 3^\circ\text{C}$. Two tests will be performed:

1. 60 mL of 20% acetic acid will be added to the reaction vessel. Once the system has equilibrated to 21°C , a syringe will be used to add 450 μL of Lewisite to the reaction vessel.
2. 60 mL of 7.0 M nitric acid will be added to the reaction vessel. Once the system has equilibrated to 21°C , a syringe will be used to add 450 μL of a 2-hour acetic acid rinse (sample HOAC-1 produced during the rinse test described in Section 3.6.3) to the reaction vessel.

For all calorimetry testing the mass of reactants will be recorded. The reactor will be stirred at a rate of approximately 200 rpm. Heat flow and temperature will be monitored until the heat flow stabilizes. The data will be reported as total heat (J) and maximum rate of heat release per unit of Lewisite in the first test and per unit of acetic acid rinse in the second test.

A mass flow meter capable of measuring low flow rates (0.01 to 1.0 mL/min) will be attached to the vent valve on the reactor. Flow rate data will be used to determine total gas volume evolved during the course of each reaction. Because the calorimeter will maintain isothermal conditions, $P \cdot V$ is constant, therefore:

Equation 1 $P_1 V_1 = P_2 V_2$

Where P_1 = atmospheric pressure
 V_1 = reactor headspace volume
 P_2 = pressure due to evolved reaction gas
 V_2 = volume of evolved reaction gas + reactor volume headspace

Equation 1 will be used to calculate the P_2 , the pressure created by the evolved reaction gas.

A Tedlar[®] bag will be used to capture any evolved gas. A Dräger MiniWarn chemical vapor detector will be used to monitor the Tedlar bag for NO and NO₂; however, if a low volume of gas is generated it may not be possible to obtain an accurate reading.

4 0 SAFETY AND ENVIRONMENTAL

The safety and environmental controls implemented by Battelle and the HMRC are discussed below

4 1 Safety

Testing will be conducted in accordance with the Battelle Columbus Operations Environment, Safety, and Health (ES&H) Management Plan. All tests will be conducted at Battelle's HMRC. Standard Operating Procedures (SOPs) have been developed to cover all procedures performed in the HMRC, including general and unique operations, personal protective equipment (PPE), CA material handling, decontamination, disposal, evacuation, and emergency response. SOPs that will be followed for this testing include SOP HMRC I-016, SOP HMRC I-034, SOP HMRC II-001 and, SOP HMRC III-07.

All HMRC technical and support personnel have been extensively trained in the requisite procedures to ensure the safe handling of hazardous and toxic substances. Battelle SOPs are internal documents and are available at the HMRC for review.

Test equipment will be set up with approved ventilation in Hazardous Materials Laboratories at the HMRC. Practice runs will be conducted to ensure that all test equipment functions properly prior to conducting a safety dry run which will be performed using CA simulant to ensure the safety of the operation and will be witnessed by a Battelle Chemical Hygiene Officer (CHO). Project specific SOPs will be prepared for every program performed at the HMRC. Before testing is initiated, project personnel are required to perform a dry run of the project specific SOP developed for the program. This dry run will help identify possible problems in the test configuration, sampling procedures, and safety protocols. Testing will begin after successful completion of the dry run and safety hazard analysis. The CHO will ensure that all approved safety procedures have been properly implemented and are being enforced.

Chemical agent operations will be conducted in accordance with the current bailment agreement between Battelle and the Government.

4 2 Environmental

Testing will be conducted in accordance with the Battelle Columbus Office (ES&H) Management Plan and the HMRC Chemical Hygiene Plan. Decontamination and disposal of laboratory wastes produced at the HMRC will be conducted in accordance with SOP HMRC I-011. The Battelle Columbus Operations (ES&H) Management Plan is the highest level document describing the general policies, committees and other means of managing (ES&H) within Battelle Columbus Operations. The HMRC has been part of the BCO and is currently a part of the BSTI. Thus, the document currently entitled BCO ES&H Management Plan describes requirements and processes that cover all of the BSTI (which includes the HMRC). The SOP HMRC I-011 describes specific waste disposal processes unique to the HMRC, but which also are compliant with Environmental requirements established for all of BSTI. The HMRC SOP also specifies requirements that are in compliance with the Bailment Agreement and Joint Army clauses.

5 0 QUALITY ASSURANCE / QUALITY CONTROL

A summary of analytical quality requirements for the sampling program is presented in Tables 3 through 12. QC results outside of the control limits will be addressed as outlined in Tables 3 through 12. All work will be performed in accordance with the CEMO and HMRC Quality System.

5 1 Data Management and Review

Samples will be tracked by chain-of-custody procedures. Laboratory record book(s) will be used to designate sample identification and track sample analysis, storage, and destruction. Verification (signature) of all data entered daily into Laboratory Record Books (LRBs) is required as well as review (signature) of all data by a reviewer. All calculations will be performed using either Atlas 8.1 chromatography software from Thermo Electron Corporation or a Microsoft Excel spreadsheet and documented as required.

5 2 Method Validation

A method detection limit study consistent with 40 CFR Part 136 Appendix B will be performed for the analysis of L1 in aqueous samples.

ICP-MS instrument detection limits for the HRA metals will be determined per Chapter One of SW-846. Initial ICP-MS operator certification for the HRA metals will be performed by the successful analysis of three blind laboratory control samples.

5 3 Controls

See Sections 3.5 and 3.6.

5 4 Calibration

See Sections 3.5 and 3.6.

5 5 Documentation

Laboratory record book(s) will be used to designate sample identification and track sample analysis, storage, and destruction. Test Performance Control Sheets (TPCS) will be used to specify and record data measurement. See Appendix B for example TPCSs. Maintenance log books will be used to document design changes or maintenance performed on instruments and test equipment. A SharePoint Site will be used as a repository for all project documentation and to provide test personnel with electronic copies of test documentation and results.

Precision

Precision is defined as the degree of mutual agreement among individual measurements made under prescribed conditions. The precision goals are included in Tables 3 through 12.

Precision will be calculated for laboratory duplicate analysis using Equation 2.

Equation 2
$$RPD = \left[\frac{|X_1 - X_2|}{\left(\frac{X_1 + X_2}{2} \right)} \right] \times 100$$

Where RPD = Relative Percent Difference
 X_1 = Analytical Result of Sample
 X_2 = Analytical Result of Sample Duplicate

Accuracy

Accuracy is the degree of agreement of a measurement to an accepted reference or true value. The accuracy will be determined from analysis of samples spiked with a known concentration. Accuracy objectives have been set and are presented in Tables 3 through 12. The formula which will be used to assess the accuracy of the laboratory QA/QC data (e.g., matrix spike analysis) is as follows:

Equation 3
$$\%R = \left(\frac{Q_{ss} - Q_{us}}{Q_s} \right) \times 100$$

Where %R = Percent recovery
 Q_{ss} = Quantity of Analyte Found in the Spiked Sample
 Q_{us} = Quantity of Analyte Found in the Unspiked Sample
 Q_s = Quantity of Added Spike

Note: For ICV/CCV, no unspiked samples, therefore $Q_{us} = 0$

Completeness

Completeness is defined as the amount of valid data from a measurement system compared to the amount that was expected under optimal normal conditions. Completeness should be 100%.

While only a single 2.0 mL or 4.0 mL sample will be collected from each TC, not all of the sample will be consumed during analysis. If the results of the initial analysis are not acceptable, the remaining portion of the sample can be analyzed, except for pH testing, which will be performed in duplicate, and VOCs, for which there will only be one sample collected.

Completeness will be reported as the percentage of all measurements judged to be valid. Every attempt will be made to ensure that all data generated will be valid data. If data appears questionable based on circumstances that occurred or were observed during either the field sampling or laboratory analyses, it will be flagged and an explanation provided.

5.6 Audits

Routine quality assurance audits, critical phase reviews and data review will be conducted by a Battelle quality assurance officer.

5 7 Process for Test Plan Changes

Changes to this test plan will be made only as required to work around infeasible test conditions or changes required by the client that are within scope. All changes will be handled as either test plan revisions with an appropriate revision number, or will be tracked as a memo attached to the test plan. All major changes will be submitted for approval by the signature list of the original test plan. Major changes include changes in test conditions that may affect how the data will be ultimately used or changes in key staff. Minor changes include changes to TPCSs, changes to test schedules caused by delays, changes in test execution, and minor changes to test conditions. Minor changes will be approved by the Test Coordinator with concurrence from the Battelle PM and documented in a memo.

5 8 Non-Conforming Data

Statistical analysis will be performed on data sets to detect outliers. All data will be included in the test report. Only data with assignable cause will be excluded from the statistical calculation of results.

6 0 REPORTING

A final report will be prepared detailing the test and test results. A hard copy and an electronic version of the report will be provided. Complete data packages consisting of all test data will be provided to TOCDF in PDF format.

7 0 REFERENCES

- 1 TOCDF LOP TE-LOP-557, Analysis of Metals by ICP-MS
- 2 SW-846 Method 9045D, Soil and Waste pH
- 3 SOP HMRC I-023-14, "Chemical Agent (CA) Receipt, Storage, Accountability and Reporting at the HMRC"
- 4 HMRC IV-055-05, "Purity and Impurity Analysis of Solutions Containing Chemical Agent by Gas Chromatography"
- 5 HMRC IV-056-11, "Operation and Maintenance of Gas Chromatography and Analysis of Solutions Containing GA, GB, GD, GF, HD, L, and VX by Gas Chromatography"
- 6 HMRC IV-067-04, "Quantitative Analysis of HD, GA, GB, GD, and VX Collected on Solid Sorbent"
- 7 ECBC-TR-531 Appendix - Standard Operating Procedure for "Multi-Residue Quantitative Analysis of HD, HN3, Lewisite and Other Arsenical Chemical Warfare Agents in Permanganate-Based Demilitarization Waste Streams"
- 8 Battelle Chemical Environmental and Materials Operations (CEMO) Quality Manual

Appendix A

Table of Samples and Analyses

TC Type	Sample Name	Sample Size	Sample Type	Analysis
GA	D-25253-S-01	2 0 mL	Sludge	HRA Metals
	D-25253-L-01	4 0 mL	Liquid	pH, density, purity, TICs, chlorobenzene, HRA metals
	D-25253-L-B	4 0 mL	Liquid Field Dup	pH, density, purity, TICs, chlorobenzene, HRA metals
	D-35248-S-01	2 0 mL	Sludge	HRA Metals
	D-35248-L-01	4 0 mL	Liquid	pH, density, purity, TICs, chlorobenzene, HRA metals
	D-29813-S-01	2 0 mL	Sludge	HRA Metals
	D-29813-L-01	4 0 mL	Liquid	pH, density, purity, TICs, chlorobenzene, HRA metals
	D-51365-S-01	2 0 mL	Sludge	HRA Metals
	D-51365-L-01	4 0 mL	Liquid	pH, density, purity, TICs, chlorobenzene, HRA metals
Lewsite	D-49221-S-01	2 0 mL	Sludge	HRA Metals
	D-49221-L-01	4 0 mL	Liquid	pH, density, purity, TICs, HRA metals
	D-49221-L-B	4 0 mL	Liquid Field Dup	pH, density, purity, TICs, HRA metals
	D-79685-S-01	2 0 mL	Sludge	HRA Metals
	D-79685-L-01	4 0 mL	Liquid	pH density purity, TICs, HRA metals
	D-79685-L-02	30 0 mL	Liquid	Solubility, rinse test, calorimetry
	D-79693-S-01	2 0 mL	Sludge	HRA Metals
	D-79693-L-01	4 0 mL	Liquid	pH, density, purity, TICs, HRA metals
	D-79697-S-01	2 0 mL	Sludge	HRA Metals
	D-79697-L-01	4 0 mL	Liquid	pH, density, purity, TICs, HRA metals
	D-79699-S-01	2 0 mL	Sludge	HRA Metals
	D-79699-L-01	4 0 mL	Liquid	pH, density, purity, TICs, HRA metals
	D-70700-S-01	2 0 mL	Sludge	HRA Metals
	D-70700-L-01	4 0 mL	Liquid	pH, density, purity, TICs, HRA metals
	D-79701-S-01	2 0 mL	Sludge	HRA Metals
	D-79701-L-01	4 0 mL	Liquid	pH, density, purity, TICs, HRA metals
	D-79703-S-01	2 0 mL	Sludge	HRA Metals
	D-79703-L-01	4 0 mL	Liquid	pH, density, purity, TICs, HRA metals
	D-79705-S-01	2 0 mL	Sludge	HRA Metals
	D-79705-L-01	4 0 mL	Liquid	pH, density, purity, TICs, HRA metals
	D-79711-S-01	2 0 mL	Sludge	HRA Metals
	D-79711-L-01	4 0 mL	Liquid	pH, density, purity, TICs, HRA metals
Transparency	D-13754-L-01	5 0 mL	Liquid	L1, L2 and L3, HRA Metals
	D-13754-G-01	1 0 L @ 200 mL/min	Air	VOCs
	D-2425-L-01	5 0 mL	Liquid	L1, L2 and L3, HRA Metals
	D-2425-G-01	1 0 L @ 200 mL/min	Air	VOCs
	D-46304-L-01	5 0 mL	Liquid	L1, L2 and L3, HRA Metals
	D-46304-G-01	1 0 L @ 200 mL/min	Air	VOCs
	D-81037-L-01	5 0 mL	Liquid	L1, L2 and L3, HRA Metals
	D-81037-G-01	1 0 L @ 200 mL/min	Air	VOCs
	D-43593-L-01	5 0 mL	Liquid	L1, L2 and L3, HRA Metals

TC Type	Sample Name	Sample Size	Sample Type	Analysis
	D-43593-G-01	1 0 L @ 200 mL/min	Air	VOCs
	D-34998-L-01	5 0 mL	Liquid	L1, L2 and L3, HRA Metals
	D-34998-G-01	1 0 L @ 200 mL/min	Air	VOCs
	D-34998-L-B	5 0 mL	Liquid Field Dup	L1, L2 and L3, HRA Metals
	D-39003-L-01	5 0 mL	Liquid	L1, L2 and L3, HRA Metals
	D-39003-G-01	1 0 L @ 200 mL/min	Air	VOCs
	D-45358-L-01	5 0 mL	Liquid	L1, L2 and L3, HRA Metals
	D-45358-G-01	1 0 L @ 200 mL/min	Air	VOCs
	D-53763-L-01	5 0 mL	Liquid	L1, L2 and L3, HRA Metals
	D-53763-G-01	1 0 L @ 200 mL/min	Air	VOCs
	D-26234-L-01	5 0 mL	Liquid	L1, L2 and L3, HRA Metals
	D-26234-G-01	1 0 L @ 200 mL/min	Air	VOCs

Appendix B

Rinse Test Procedure

Two rinse tests, designated A and B, will be performed in parallel. The addition of the 20% w/v solution of acetic acid, where noted, will be performed two ways

- Rinse Test A – add 1350 mL of 20% acetic acid
- Rinse Test B – add 810 mL of water and then add 540 mL of 50% acetic acid

Note The procedure detailed below is identical in content to the one supplied in the Statement of Work, however, some step numbers have been changed to letters for ease of reading. No steps are missing

- 1) **PIPETTE** 10.0 mL of Lewisite onto four (4) coupons of ton container material placed in the base of a 2 L polycarbonate wide mouth container. Ensure each coupon is covered with agent. **SEAL** container following addition of agent. Prepare in duplicate
 - 2) **ALLOW** to stand for twenty-four (24) hours
 - 3) **ADD** 1350 mL of a 20% w/v solution of Acetic Acid (HOAC), **SEAL** container and **LABEL** container HOAC-1
 - 4) **PLACE** the container on a ball mill. Optimum rotation two revolutions per min
 - 5) **AT** the two (2) hour mark **STOP** container rotation and **REMOVE** 400 mL and one (1) coupon for analysis, **REMOVE** and **SEGREGATE** the three remaining coupons for transfer to second (2nd) container per Step 12. Once all required material has been removed **RESUME** container rotation
 - a **SEGREGATE** 10 mL of HOAC-1 solution for use in generating HOAC-2 at Step 12
 - b **SEGREGATE** 10 mL of HOAC-1 solution for use in generating HOAC-1/HNO₃-1 at Step 14
 - 8) **ANALYZE** the remaining 380 mL of solution for
 - L₁, L₂, L₃ from the HOAC-1 and
 - LL₁, L₂, L₃ Arsenic (As) and Mercury (Hg) as an extraction from the coupon
-

- 9) **AT** the four (4) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis. Once all required material has been removed **RESUME** container rotation. **ANALYZE** for
- L₁, L₂, and L₃ from the HOAC-1 solution
- 10) **AT** the six (6) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis. **ANALYZE** for
- L₁, L₂, and L₃ from the HOAC-1
- 11) **DECANT** the remaining HOAC-1 solution (approximately 160 mL) and **ARCHIVE** the solution
-
- 12) **ADD** 10.0 mL of the two (2) hour solution, HOAC-1, segregated in step 5, along with the remaining three (3) coupons, segregated in step 5, to a new 2nd container
- 13) **ADD** 1350 mL of a 20% w/v solution of Acetic Acid (HOAC) to the 2nd container generating HOAC-2. **SEAL** the container and **LABEL** as HOAC-2
- 14) **ADD** 10.0 mL of the two (2) hour solution, HOAC-1, segregated in step 5, to a 3rd container
- 15) **ADD** 1350 mL of a 7.0 M Nitric Acid solution to the 3rd container with the 10.0 mL of the HOAC-1 generating HOAC-1/HNO₃-1. **SEAL** the container and **LABEL** as HOAC-1/HNO₃-1
- 16) **ROTATE** the containers on ball mills at ~2 rpm
- 17) **AT** the two (2) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis from each container (HOAC-2 and HOAC-1/HNO₃-1). Each 400 mL sample is to be tagged and bottled separately for chain of custody and analysis purposes
- a **REMOVE** and **SEGREGATE** the 3 coupons from the 2nd container HOAC-2 for transfer to the 4th container per Step 24. Store locked in the fume hood or 4±3°C overnight
 - b Once the required material has been removed from each container **RESEAL** the container and **RESUME** container rotation
 - c **SEGREGATE** 10 mL of HOAC-2 solution and store at 4±3°C overnight prior to addition to the 3 coupons at Step 24 (used to generate HOAC-3 at Step 25)
 - d **SEGREGATE** 10 mL of HOAC-2 solution and store at 4±3°C overnight for use in generating HOAC-1/HNO₃-2 at Step 27

e **ANALYZE** the remaining 380 mL HOAC-2 and 400 mL of HOAC-1/HNO3-1 solutions for

- o L₁, L₂, and L₃

21) **AT** the four (4) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis from each container (HOAC-2 and HOAC-1/HNO3-1) Once the required material has been removed from each container **RESEAL** the container and **RESUME** container rotation **ANALYZE** the HOAC-2 and the HOAC-1/HNO3-1 for

- o L₁, L₂, and L₃

22) **AT** the six (6) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis from each container **ANALYZE** the HOAC-2 and the HOAC-1/HNO3-1 for

- o L₁, L₂, and L₃

23) **DECANT** the remaining HOAC-2 and HOAC-1/HNO3-1 solutions (approximately 160 mL each) from each container and **ARCHIVE** each solution separately

THIS WILL BE THE END OF THE FIRST DAY OF TESTING

24) **ADD** 10 0 mL of the two (2) hour solution, HOAC-2 segregated at Step 17, along with the remaining three (3) coupons segregated in step 17 to a separate 4th container

25) **ADD** 1350 mL of a 20% w/v solution of Acetic Acid to the 4th container with the coupons generating solution HOAC-3 **SEAL** the container and **LABEL** as HOAC-3

26) **ADD** 10 0 mL of the two (2) hour solution, HOAC-2, segregated at step 17, to a 5th container

27) **ADD** 1350 mL of a 7 0 M Nitric Acid solution to the 5th container with the 10 0 mL of the HOAC-2 generating solution HOAC-2/HNO3-2 **SEAL** the container and **LABEL** as HOAC-2/HNO3-2

28) **ROTATE** the containers on a ball mill

29) **AT** the two (2) hour mark **STOP** container rotation and remove 400 mL for analysis from each container Each 400 mL sample is to be tagged and bottled separately for chain of custody and analysis purposes

a **REMOVE** and **SEGREGATE** the 3 coupons from the 4th container for use when generating HOAC-3/HNO3-3 beginning at Step 35

- b Once the required material has been removed from each container **RESEAL** the container and **RESUME** container rotation
 - c **SEGREGATE** 10 mL of HOAC-3 solution for use in generating HOAC-3/HNO3-3 at step 35
 - d **ANALYZE** the remaining 390 mL HOAC-3 and the HOAC-2/HNO3-2 solutions for
 - o L₁, L₂, and L₃
-

- 32) **AT** the four (4) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis from each container. Once the required material has been removed from each container **RESEAL** and **RESUME** container rotation. Each 400 mL sample is to be tagged and bottled separately for chain of custody and analysis purposes. **ANALYZE** the HOAC-3 and the HOAC-2/HNO3-2 for
 - o L₁, L₂, and L₃
 - 33) **AT** the six (6) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis from each container. Each 400mL sample is to be tagged and bottled separately for chain of custody and analysis purposes. **ANALYZE** the HOAC-3 and the HOAC-2/HNO3-2 for
 - o L₁, L₂, and L₃
 - 34) **DECANT** the remaining solution (approximately 160 mL each) from each container and **ARCHIVE** each solution separately
-

- 35) **ADD** 10.0 mL of the two (2) hour solution (HOAC-3), segregated in Step 29, and the remaining three (3) coupons to a 6th container
- 36) **ADD** 1350 mL of 7.0 M Nitric Acid to container 6 generating HOAC-3/HNO3-3. **SEAL** the container and **LABEL** as HOAC-3/HNO3-3
- 37) **ROTATE** the containers on a ball mill
- 38) **AT** the two (2) hour mark **STOP** container rotation and **REMOVE** 400 mL and one coupon for analysis
 - a **REMOVE** and **SEGREGATE** the 2 remaining coupons from the 6th container for use when generating HOAC-3/HNO3-4 at Step 43. Store locked in the fume hood or 4±3°C overnight
 - b **SEGREGATE** 10 mL of sample and store at 4±3°C overnight for use in generating HOAC-3/HNO3-4 at Step 43

- c Once the required material has been removed from the container **RESEAL** and **RESUME** container rotation
 - d **ANALYZE** the HOAC-3/HNO3-3 and extracted coupon for
 - o L₁, L₂, L₃,
 - o As and
 - o Hg
-

- 40) **AT** the four (4) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis Once the required material has been removed from each container **RESEAL** and **RESUME** container rotation **ANALYZE** the HOAC-3/HNO3-3 for
- o L₁, L₂, L₃
 - o As and
 - o Hg
- 41) **AT** the six (6) hour mark **STOP** the container rotation and **REMOVE** 400 mL for analysis **ANALYZE** the HOAC-3/HNO3-3 for
- o L₁, L₂, L₃,
 - o As and
 - o Hg

- 42) **DECANT** the remaining solution (approximately 160 mL) and **ARCHIVE**

THIS WILL BE THE END OF THE SECOND DAY OF TESTING

- 43) **ADD** 10 0 mL of the two (2) hour solution (HOAC-3/HNO3-3) and the remaining two (2) coupons, segregated in step 38 to a 7th container
- 44) **ADD** 1350 mL of 7 0 M Nitric Acid generating HOAC-3/HNO3-4 **SEAL** the container and **LABEL** as HOAC-3/HNO3-4
- 45) **ROTATE** the container on a ball mill
- 46) **AT** the two (2) hour mark **STOP** container rotation and **REMOVE** 400 mL and one coupon for analysis
- a **REMOVE** and **SEGREGATE** the remaining coupon from the 7th container for use when generating HOAC-3/HNO3-5 at Step 51
 - b **SEGREGATE** 10 mL of sample for use in generating HOAC-3/HNO3-5 at Step 51
 - c Once the required material has been removed from the container **RESEAL** and **RESUME** container rotation

d **ANALYZE** the remaining 390 mL HOAC-3/HNO3-4 and extracted coupon for

- L₁, L₂, L₃,
 - As and
 - Hg
-

48) **AT** the four (4) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis. Once the required material has been removed from each container **RESEAL** and **RESUME** container rotation. **ANALYZE** the HOAC-3/HNO3-4 for

- L₁, L₂, L₃,
- As and
- Hg

49) **AT** the six (6) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis. **ANALYZE** the HOAC-3/HNO3-4 for

- L₁, L₂, L₃,
- As and
- Hg

50) **DECANT** the remaining solution (approximately 160 mL) from the container and **ARCHIVE** the solution

51) **ADD** 10.0 mL of the two (2) hour solution (HOAC-3/HNO3-4) and the remaining one (1) coupon, segregated in Step 46 to an 8th container

52) **ADD** 1350 mL of 7.0 M Nitric Acid generating HOAC-3/HNO3-5. **SEAL** the container and **LABEL** as HOAC-3/HNO3-5

53) **ROTATE** the container on a ball mill

54) **AT** the two (2) hour mark **STOP** container rotation and **REMOVE** 400 mL and the last coupon for analysis

a **SEGREGATE** 10 mL of the solution for the generation of the first triple rinse solution beginning at Step 59

b **ANALYZE** the remaining 390 mL HOAC-3/HNO3-5 and extracted coupon for

- L₁, L₂, L₃,
 - As and
 - Hg
-

- 56) **AT** the four (4) hour mark **STOP** container rotation and remove 400 mL for analysis. Once the required material has been removed from the container **RESEAL** and **RESUME** container rotation. **ANALYZE** the HOAC-3/HNO3-5 for
- L₁, L₂, L₃,
 - As and
 - Hg
- 57) **AT** the six (6) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis. **ANALYZE** the HOAC-3/HNO3-5 for
- L₁, L₂, L₃,
 - As and
 - Hg
- 58) **DECANT** the remaining solution (approximately 160 mL) from the container and **ARCHIVE** the solution
-
- 59) **ADD** 100 mL of the two (2) hours HOAC-3/HNO3-5 solution to a 9th container and **ADD** 1350 mL of water generating the first triple rinse (TR-1). **SEAL** and **LABEL** the container TR-1
- 60) **ROTATE** the container on a ball mill
- 61) **AT** the one (1) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis
- a **SEGREGATE** 100 mL for use in generating the second triple rinse (TR-2) beginning at step 65
 - b **ANALYZE** the remaining 390 mL solution for
- L₁, L₂, L₃,
 - As and
 - Hg
- 64) **DECANT** the remaining (960 mL) TR-1 solution and **ARCHIVE**
-
- 65) **ADD** 100 mL of the first triple rinse (TR-1) to a 10th container and **ADD** 1350 mL of water generating the second triple rinse (TR-2). **SEAL** the container and **LABEL** the container TR-2
- 66) **ROTATE** the container on suitable device (refer to step 4 for guidance)
- 67) **AT** the one (1) hour mark **STOP** container rotation and **REMOVE** 400 mL for analysis

- a **SEGREGATE** 10 ml for use in generating the third triple rinse (TR-3) beginning at step 71
- b **ANALYZE** the remaining 390 mL of solution for
 - L1, L2, L3,
 - As and
 - Hg

70) **DECANT** the remaining 960 mL TR-2 solution and **ARCHIVE**

71) **ADD** 10 0 mL of the second triple rinse solution (TR-2) to an 11th container and **ADD** 1350 mL of water generating the third triple rinse solution, TR-3

72) **ROTATE** the container on suitable device (refer to step 4 for guidance)

73) **AT** the one (1) mark **STOP** container rotation and **REMOVE** 400 mL for analysis and **ANALYZE** for

- L1, L2, L3,
- As and
- Hg

74) **DECANT** the remaining 960 mL TR-3 solution and **ARCHIVE**

Appendix C

Example Test Performance Control Sheet

Test Type pH
Date
Operators

Water Lot
No
pH Meter ID
pH Probe
ID

Cal Standard ID	pH Value

Sample ID	Agent	Sample Weight (g)	Water Volume (mL)	Mix Time (hh mm)	Measurement Time (hh mm)	pH Value

Appendix D
Comments/Changes

Malloy, Thomas

From Williams Larry [lwilliams@egginc.com]
Sent Tuesday, June 16, 2009 2:53 PM
To Malloy, Thomas
Subject RE: TC Sample Analysis Test Plan

Sounds good, let's take the test plan to final

From Malloy, Thomas [mailto:MalloyT@BATTELLE.ORG]
Sent Tuesday, June 16, 2009 12:50 PM
To Williams, Larry
Cc Clark, Jim A, Whittington, Katherine F
Subject RE: TC Sample Analysis Test Plan

Larry

This is just a standard statement included in our test plan that clarifies the source of the agent for calibration and spiking standards. Since we already have agent available, you don't need to do anything. However, we still need to ask permission from ECBC to use it.

The only deviations will be non-technical items that are specific to the CAL operations, such as how the agent is handled or references to specific lab room numbers at the CAL. Our SOP will include our own agent handling specifications, but all of the technical and QC requirements will match TE-LOP-557 and TE-LOP-584.

Let me know if you need any additional information.

Tom

From Williams, Larry [mailto:lwilliams@egginc.com]
Sent Tuesday, June 16, 2009 2:39 PM
To Malloy, Thomas
Cc Clark, Jim A
Subject RE: TC Sample Analysis Test Plan

Tom, I have just two questions about the Test Plan.

On page 4, what is meant by "Agent for the preparation of calibration and spiking standards will be provided by TOCDF through ECBC or transferred to this task from other TOCDF projects"?

Throughout the document, it is stated that the metals analysis will be **based** on TE-LOP-557 and TE-LOP-584. What are the deviations?

From Malloy, Thomas [mailto:MalloyT@battelle.org]
Sent Tuesday, June 09, 2009 11:35 AM
To Williams, Larry
Cc Whittington, Katherine F, Balaban, Elizabeth A
Subject TC Sample Analysis Test Plan

Larry

Here is the latest revision of our draft test plan for the TC sample analysis. We have corrected the report due date on the schedule. Also, we have updated the Rinse Test procedure (Section 3.6.2) based on our earlier

6/17/2009

discussions Table 2 and Appendix A have been updated to reflect the changes to the Rinse Test procedure. Some additional verbiage describing the VOC analysis was added to Section 3.5.8. These are the only changes.

Regards,

Tom

6/17/2009

August 20, 2009

To Kathy Whittington

From Tom Malloy

Subject Change Memo for Test Plan for Ton Container Sample Analysis

Kathy,

The below changes to the Test Plan for Ton Container Sample Analysis need to be implemented

Section 3.5.1

Based on information recently reviewed regarding the hydrolysis of Lewisite, the use of EPA Method 8045D for determining pH is not appropriate for this agent. According to Munro et al (1999), hydrolysis is rapid resulting in the formation of the water-soluble 2-chlorovinyl arsonous acid (CVAA). Also, according to Haigh (2007), 1 mole of Lewisite undergoes a fast reaction with 2 moles of water to form 1 mole of CVAA and 2 moles of HCl; this hydrolysis reaction is quantitative. While no half-life or kinetic data is available in the literature, the above information indicates that the use of Method 8045D, with the 1:1 mixing of sample and water, is not appropriate – particularly since the formation of HCl will result in pH measurement that is biased low. Additionally, a rapid hydrolysis reaction may result in the evolution of significant amounts of heat and therefore present a safety concern.

Lewisite sample pH will instead be measured directly using the microprobe purchased for this project. The initial pH reading will be recorded and, after allowing the reading to stabilize, the final pH reading will be recorded. If the pH reading does not stabilize, the range of pH readings will be recorded.

Section 3.6.2

During Safety Dry Run testing, the simulant DMMP reacted with the polycarbonate sample bottle, resulting in loss of test liquid from the bottle. This raised concerns about compatibility of polycarbonate with Lewisite. A review of polycarbonate chemical compatibility indicated that polycarbonate is not compatible with chlorinated solvents. Based on this, the decision was made to replace the polycarbonate bottles with equivalent glass bottles.

Section 3.6.3

A Tedlar bag will be used to collect evolved gases during the calorimetry tests. A gas-tight syringe will then be used to measure the volume of the evolved gas contained in the bag. This will provide a simple and accurate means of determining gas volume that will be used to determine pressure. The gases will then be injected from the syringe into the MiniWarn gas monitor to measure NO and NO₂.

An additional test was added:

A test will be performed to determine the solubility of sludge from Lewisite ton containers in three different solvents. Samples will be prepared in a ratio of 1 g of sludge to 125 mL of solvent. The three solvents that will be tested are water, 20% acetic acid and 7 M nitric acid. Sample D-79711-S-01 is proposed for the solubility testing. This sample contained a Hg content of 438,000 mg/Kg. Based upon the photo shown in Figure 1, there appears to be sufficient material to collect six 0.1 g samples. Table 1 shows the test matrix. QC samples will be used to create blank samples and laboratory control samples.

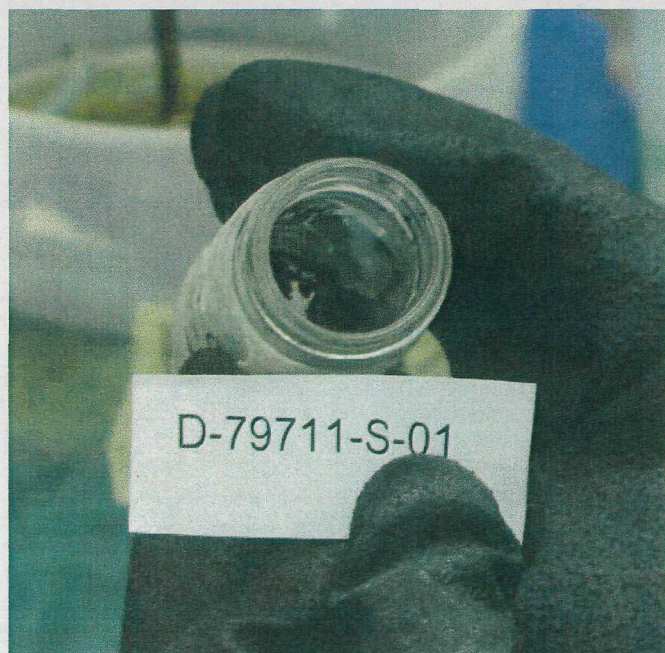


Figure 1. Photo of sample D-79711-S-01

Table 1. Test Matrix

Sample No.	Sample Type	Weight (g)	Solvent	Solvent Vol (mL)	Analyses
1	Sludge	0.1	Water	12.5	% Solids, L, As, Hg
2	Sludge	0.1	Water	12.5	% Solids, L, As, Hg
3	QC	NA	Water	12.5	% Solids, L, As, Hg
4	Sludge	0.1	Acetic Acid	12.5	% Solids, L, As, Hg
5	Sludge	0.1	Acetic Acid	12.5	% Solids, L, As, Hg
6	QC	NA	Acetic Acid	12.5	% Solids, L, As, Hg
7	Sludge	0.1	Nitric Acid	12.5	% Solids, L, As, Hg
8	Sludge	0.1	Nitric Acid	12.5	% Solids, L, As, Hg
9	QC	NA	Nitric Acid	12.5	% Solids, L, As, Hg

Approximately 0.1 g of sample will be weighed into a 30 mL polypropylene container. 12.5 mL of test solvent will be added and the solution mixed end-over-end for at least 2 hr using a rotary mixer. Based on an As concentration of 146,000 mg/Kg the Lewisite content of D-79711-S-01 cannot be above 40%, therefore, the prepared solutions will not contain Lewisite above 3.2 mg/mL. This will allow samples to be treated as dilute agent solutions.

Samples will first be filtered through a glass fiber filter and the filters dried in an oven at 103 - 105°C consistent with Standard Methods 2540D "Total Suspended Solids Dried at 103 - 105°C". The amount of solids retained on the filter will be determined gravimetrically. This method should have a sensitivity of about 15 mg/L.

The filtrate from each sample will then be analyzed for L1 as its ethanethiol derivative by GC/MS. The analytical method discussed in Test Plan Section 3.5.7 will be used. The filtrate from each sample will also be analyzed for As and Hg by ICP/MS using the analytical method discussed in Test Plan Section 3.5.9. If the L1 content is below the drinking water level of 80 µg/L the samples will be handled as drinking waters, otherwise, samples will be handled as RDTE dilute solutions and shipped accordingly to Battelle labs at King Ave. for digestion and metals analysis in Building 20-2. For each sample, the amount of As and Hg remaining on the filter paper will be determined by mass balance using data from the initial analysis of the sludge sample and data from the amount of As and Hg detected in the sample filtrate.

Thomas A. Malloy IV

8/20/2009

Thomas A. Malloy IV

Katherine Whittington

8/20/2009

Katherine Whittington

References

"The Sources, Fate, and Toxicity of Chemical Warfare Agent Degradation Products", Munro et al., Volume 107, Number 12, December 1999, Environmental Health Perspectives

Executive Summary of "Chemical Warfare Agents and their Hydrolysis Products from the US EPA Standardized Analytical Methods and GC-MS Analytical Method for the Analysis of Chemical Warfare Agent Degradation Products Listed in the EPA Standardized Analytical Methods", Haigh, Theodore A., Prepared for NEMC Conference, Cambridge, MA, Aug. 20-24, 2007

September 2, 2009

To Kathy Whittington

From Tom Malloy

Subject Change Memo for Test Plan for Ton Container Sample Analysis

Kathy,

The below tests will supplement or replace Rinse Test steps 5 - 74 discussed in Section 3.6.2 of the Test Plan and shown in Appendix B. Testing procedure changes performed on August 25, 2009 have been captured in the project Laboratory Record Book (LRB TC-1)

Two events on August 25, 2009 led to these changes. First, addition of 20% acetic acid to the Lewisite and ton container coupons per Step 3 in Appendix B resulted in a brown, viscous precipitate which coated the bottom and sides of the bottle, only minimal solubilization of the Lewisite by the acetic acid was apparent. Discussion of this result led to the decision, initiated by Larry Williams (EG&G), to skip from Step 5 to Step 35, bypassing additional acetic acid rinsing and going directly to rinsing with 7.0 M nitric acid.

Second, as part of this initial testing change, one TC coupon and all but 10 mL of acetic acid rinse were removed from the bottles holding samples HOAC-1A and HOAC-1B. When the 7.0 M nitric acid was added to the residual Lewisite and three TC coupons remaining in the HOAC-1A bottle, the nitric acid reacted with the TC coupons forming a significant amount of NO_x gas and foam. The foam boiled over the edge of the 2 L bottle. This test was then terminated. Discussion with Larry Williams resulted in the Test Plan change outlined below. This change was captured in a change memo to SOP HMRC-X-243 and signed by all Battelle staff prior to starting work.

I Sample HOAC-1A

- 1 Transfer sample to a new 2 L glass bottle
- 2 Add remaining 7.0 M nitric acid solution (~600 mL) to the sample
- 3 Archive bottle
- 4 Archive the sample coupons separately in zip lock bags

II Sample HOAC-1B

Step A

- 1 Remove two coupons and place in zip-lock bags
- 2 Slowly add 1350 mL of 7.0 M nitric acid (~100 mL at a time). Watch for a chemical reaction and the evolution of brown gas. Monitor temperature.
- 3 Rotate solution for 2 hr on ball mill at ~ 2 rpm
- 4 Remove a subsample and analyze for Lewisite, As and Hg
- 5 Remove the coupon and analyze for Lewisite, As and Hg

Step B

- 1 Remove 10 mL of sample from Step II A and add to a new 2 L polycarbonate bottle Do **not** add the coupon
- 2 Add 1350 mL of DI water
- 3 Rotate for 1 hr on a ball mill at ~ 2 rpm
- 4 Remove a subsample and analyze for Lewisite, As and Hg

Step C

- 1 Remove 10 mL of sample from Step II B and add to a new 2 L polycarbonate bottle
- 2 Add 1350 mL of DI water
- 3 Rotate for 1 hr on a ball mill at ~ 2 rpm
- 4 Remove a subsample and analyze for Lewisite, As and Hg

Step D

- 1 Remove 10 mL of sample from Step II C and add to a new 2 L polycarbonate bottle
- 2 Add 1350 mL of DI water
- 3 Rotate for 1 hr on a ball mill at ~ 2 rpm
- 4 Remove a subsample and analyze for Lewisite, As and Hg

III Nitric Acid - Lewisite Rinse Test

Step A

- 1 Spike 10 mL of Lewisite onto a new tin container coupon in a 2 L glass bottle Prepare in duplicate
- 2 Add 135 mL of **3.0 M** nitric acid to one container Add acid slowly and monitor the reaction Watch for a chemical reaction and the evolution of brown gas Monitor temperature This is sample L-HNO3-3
- 3 Add 135 mL of **7.0 M** nitric acid to the second container Add acid slowly and monitor the reaction Watch for a chemical reaction and the evolution of brown gas Monitor temperature This is sample L-HNO3-7
- 4 Rotate solution for 2 hr on ball mill at ~ 2 rpm
- 5 Remove a subsample and analyze for Lewisite, As and Hg

Step B

- 1 Remove 10 mL of sample L-HNO3-3 and add to a new polycarbonate bottle Remove 10 mL of sample L-HNO3-7 and add to a second new polycarbonate bottle
- 2 Remove the coupons and add one coupon to each sample, keeping coupons with associated liquid samples
- 3 Add 1350 mL of DI water to each bottle
- 4 Rotate for 1 hr on a ball mill at ~ 2 rpm
- 5 Remove subsamples and analyze for Lewisite, As and Hg

Step C

- 1 Remove 10 mL of each sample from Step III B and add to two new polycarbonate bottles
- 2 Remove the coupons and add one coupon to each sample, keeping coupons with associated liquid samples
- 3 Add 1350 mL of DI water to each bottle
- 4 Rotate for 1 hr on a ball mill at ~ 2 rpm
- 5 Remove subsamples and analyze for Lewisite, As and Hg

Step D

- 1 Remove 10 mL of each sample from Step III C and add to two new polycarbonate bottles
- 2 Remove the coupons and add one coupon to each sample, keeping coupons with associated liquid samples
- 3 Add 1350 mL of DI water to each bottle
- 4 Rotate for 1 hr on a ball mill at ~ 2 rpm
- 5 Remove subsamples and analyze for Lewisite, As and Hg
- 6 Remove the coupons and analyze for Lewisite, As and Hg

Section 3 6 3 – Reaction Calorimetry Tests

The reaction of 450 μ L of sample HOAC-1 and 60 mL of 7 0 M nitric acid was replaced with the reaction of 450 μ L of neat Lewisite and 60 mL of 3 0 M nitric acid

Thomas A Malloy IV
Thomas A Malloy IV

9/2/2009

Katherine Whittington
Katherine Whittington

9/2/2009

**Addendum To Final Report
For
Ton Container Sample Analysis**

November 17, 2009

Prepared By
Thomas A Malloy IV
Katherine Whittington

Hazardous Materials Research Center
Battelle
505 King Ave
Columbus, OH 43201

1 0 INTRODUCTION

After review of the results from the ton container rinse testing, reported in the Final Report For Ton Container Sample Analysis, a second rinse test was requested by EG&G, DMI

The major differences between this second rinse test and the previous rinse test are

- 1 Previously, after nitric acid was added to the jars containing the Lewisite and TC coupon, the nitric acid / Lewisite / coupon mixture was allowed to sit overnight For this second rinse test, the nitric acid contacted the Lewisite and TC coupon for shorter time period, either 2 hr or 4 hr
- 2 Previously, samples were transferred to new bottles for each rinse For this second rinse test, the same jar was used throughout, better mimicking actual ton container rinsing
- 3 For this second rinse test, Lewisite ton container sludge was added to the jar along with Lewisite liquid and the TC coupon prior to nitric acid addition Sludge was not added for the previous rinse test
- 4 For this second rinse test, after the final rinse, the interior of the jars was rinsed and analyzed for Lewisite, As and Hg The jars were not rinsed for the previous rinse test

2 0 PROCEDURES AND RESULTS

2 1 Procedures

Two ton container (TC) coupons were weighed on a calibrated balance The width and thickness of each coupon was also recorded to the nearest mm using a ruler One ton container coupon was placed in each of the two 250 mL glass bottles Sludge from ton container D-79703-S-01, containing high levels of Hg, was added to each bottle with the exact weight recorded One mL of Lewisite from ton container D-79685-L-02 was then placed on top of each coupon Table 1 shows coupon, agent and sludge data for this testing, including initial coupons weights, diameter and thickness The sludge and agent were added the day prior to the rinse test to allow additional interaction of the agent with the TC metal (see Figure 1) Samples associated with Jar 1 are part of the 2 hr Nitric Acid Rinse Test Samples associated with Jar 2 are part of the 4 hr Nitric Acid Rinse Test

Table 1 Sample Preparation Data

Jar ID	Sample ID	Coupon Wt (g)	Coupon Diam (mm)	Coupon Thickness (mm)	L Sludge Wt (g)	L Liquid Wt (g)	Lewisite Vol (mL)
1	Coupon 1	43 3533	26	10	0 2375	1 8610	1
2	Coupon 2	43 0642	26	10	0 2348	1 8907	1

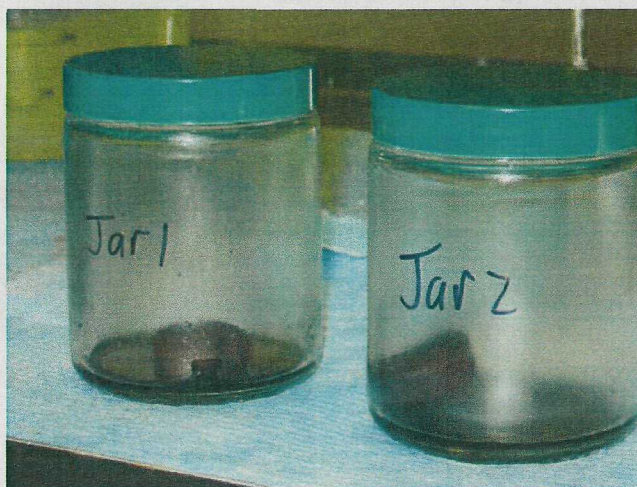


Figure 1. Jar 1 and Jar 2 the day after Lewisite liquid and sludge addition. The jars appear "foggy" due to a protective coating on the glass.

135 mL of 3.0 M HNO_3 (pH 0 as measured with broad range pH paper) was then added to each bottle. No NO_x production or acid reaction with the coupons (foaming) was initially observed. After all of the acid was added to each bottle, one bottle was rotated for 2 hr at ~ 2 rpm on a ball mill and the other bottle was rotated for 4 hr. See Table 2 for information regarding sample preparation and identification. No reaction was observed for Coupon 1 during contact with the nitric acid but some bubbles formed on the surface of Coupon 2 by the end of the 4 hr contact time. After each time period (2 hr and 4 hr), all of the acid was removed from each bottle and sub-samples were collected and quantitatively analyzed for L1, As and Hg. L2 was qualitatively analyzed by its peak area.

Table 2. Sample Preparation and Identification Information

Jar ID	Starting Material	Material Amt.	Solution Type	Solution Vol (mL)	Rinse / Ext. Time (Hr)	Sample ID
1	L Sludge	0.2375 g	3.0 M Nitric Acid	135	2	2HR-HNO3
	L Liquid	1.8610 g				
2	L Sludge	0.2348 g	3.0 M Nitric Acid	135	4	4HR-HNO3
	L Liquid	1.8907 g				
1	2HR-HNO3	1.0 mL	DI Water	135	1	2HR-DI-R1
2	4HR-HNO3	1.0 mL	DI Water	135	1	4HR-DI-R1
1	2HR-DI-R1	1.0 mL	DI Water	135	1	2HR-DI-R2
2	4HR-DI-R1	1.0 mL	DI Water	135	1	4HR-DI-R2
1	2HR-DI-R2	1.0 mL	DI Water	135	1	2HR-DI-R3
2	4HR-DI-R2	1.0 mL	DI Water	135	1	4HR-DI-R3
A	Coupon 1	1	1% Ethane Thiol/TMP	25	0.25	L-COUPON 1
B	Coupon 2	1	1% Ethane Thiol/TMP	25	0.25	L-COUPON 2
C	Coupon 3	1	1% Ethane Thiol/TMP	25	0.25	L-COUPON 3

Jar ID	Starting Material	Material Amt.	Solution Type	Solution Vol (mL)	Rinse / Ext. Time (Hr)	Sample ID
1	Empty Jar 1	NA	1% Ethane Thiol/TMP	25	0.5	L-JAR 1
2	Empty Jar 2	NA	1% Ethane Thiol/TMP	25	0.5	L-JAR 2
D	Coupon 1	1	0.1 M Nitric Acid	25	0.5	M-COUPON 1
E	Coupon 2	1	0.1 M Nitric Acid	25	0.5	M-COUPON 2
F	Coupon 3	1	0.1 M Nitric Acid	25	0.5	M-COUPON 3
1	Empty Jar 1	NA	0.1 M Nitric Acid	25	0.5	M-JAR 1
2	Empty Jar 2	NA	0.1 M Nitric Acid	25	0.5	M-JAR 2

NA = not applicable

After removal of nitric acid from the jars, 1.0 mL of the nitric acid rinse was then returned to the appropriate jar. The coupon remained in place. DI Rinse 1 was performed by adding 135 mL of de-ionized (DI) water rinse to each jar and the jar rotated for 1 hr at ~2 rpm. All of DI Rinse 1 was then removed from each jar. Two additional rinse steps were performed for each sample as follows:

- DI Rinse 2 = 1.0 mL of DI Rinse 1 plus TC coupon and 135 mL of DI water in the original container, rotated for 1 hr at ~2 rpm. All of DI Rinse 2 was then removed from each jar.
- DI Rinse 3 = 1.0 mL of DI Rinse 2 plus TC coupon and 135 mL of DI water in the original container, rotated for 1 hr at ~2 rpm. All of DI Rinse 3 was then removed from each jar.

See Table 2 for information regarding sample preparation and identification. All three water rinses were quantitatively analyzed for L1, As and Hg. L2 was qualitatively analyzed by its peak area. Photos of all samples are shown in Figure 2, Figure 3 and Figure 4.



Figure 2. 2HR-HNO₃ sample (left) and 4HR-HNO₃ sample (right) after removal from Jar 1 and Jar 2, respectively.

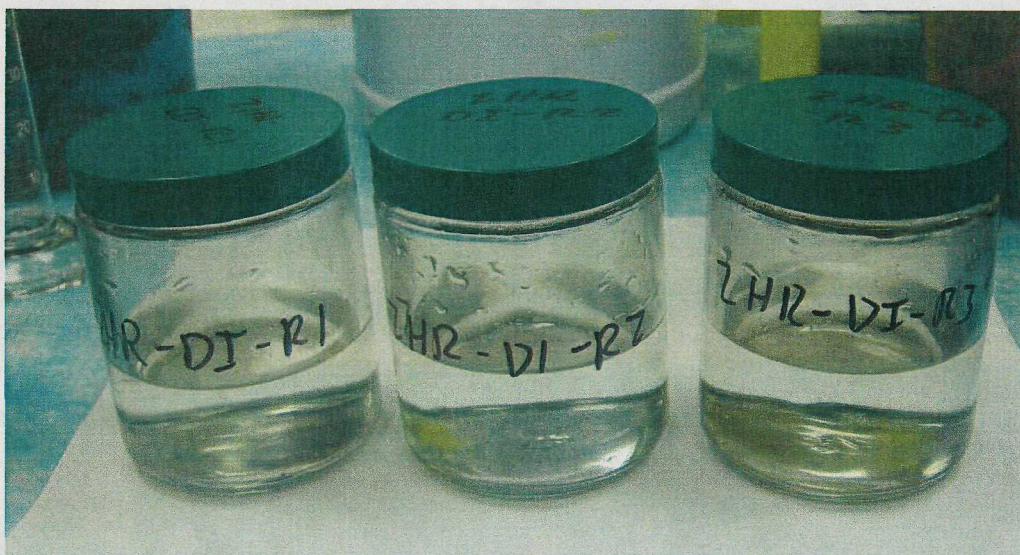


Figure 3. Samples 2HR-DI-R1 (left), -R2 (mid) and -R3 (right) after removal from Jar 1.



Figure 4. Samples 4HR-DI-R1 (left), -R2 (mid) and -R3 (right) after removal from Jar 2.

Following the last rinse, the TC coupons and final rinsate were removed from the jars. Each coupon was placed in a separate 50 mL jar (Jar A and Jar B) in 25 mL of 1% ethane thiol in 1,2,4-trimethylpentane (TMP) to extract and derivatize residual Lewisite. A clean coupon (Coupon 3) was placed in Jar C, spiked with 2.5 μg of Lewisite and also extracted. Sample extracts were identified as L-COUPON 1, L-COUPON 2 and L-COUPON 3, respectively (see Table 2).

Each coupon was then placed in a separate 50 mL jar (Jar D and Jar E) along with 25 mL of 0.1 N HNO_3 to extract residual As and Hg. Coupon 3 was placed in Jar F, spiked with 1.0 μg of As and 1.0 μg Hg and also extracted. The coupons and acid solution were heated to $\sim 50^\circ\text{C}$ for 30 min to enhance extraction. Sample extracts were identified as M-COUPON 1, M-COUPON 2 and M-COUPON 3, respectively (see Table 2).

Next, the empty jars were rinsed with 25 mL of 1% ethane thiol in TMP to remove any residual Lewisite from the jar surface. Jars were rotated for 15 min at ~2 rpm and then allowed to stand for an additional 15 min. After the 25 mL of TMP solution was removed, both jars were rinsed with 0.1 N HNO₃ to remove any residual As and Hg. Jars were rotated for 15 min at ~2 rpm and then allowed to stand for an additional 15 min. Coupon extracts and bottle rinsates were quantitatively analyzed for L1, As and Hg and L2 was qualitatively analyzed. Figure 5 shows Jar 1 and Jar 2 following extraction for As and Hg analysis. It was noted that residual material remained adhered to the walls of the jar and on bottom. In an effort to remove this material an additional 135 mL of fresh 3.0 M nitric acid was added to Jar 1. The jar was rotated at ~2 rpm for 24 hr. As can be seen in Figure 6, not all residual material was removed. Additionally, fine particulates formed during this rinse. The particulates floated on the surface of the nitric acid during rinsing but would sink to the bottom of the jar if disturbed.



Figure 5. Jar 1 (left) and Jar 2 (right) after As and Hg extraction.

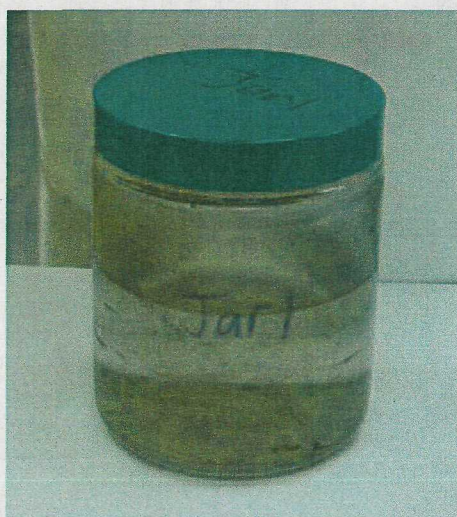


Figure 6. Jar 1 following additional 24 hr rinse with 3.0 M nitric acid.

Once all samples were collected the weight of each coupon and dimensions were then determined. Table 3 shows the initial and final coupon measurements. No changes in the diameter or thickness were measured. A slight decrease in weight was noted for each coupon: 0.34% for Coupon 1 and 0.51% for Coupon 2, indicating minor reaction between the nitric acid and coupon material, with longer contact time resulting in greater metal loss. See Figure 7 for photos of Coupon 1 and Coupon 2 after the final DI water rinse was completed.



Figure 7. Coupon 1 (left) and Coupon 2 (right) after final DI water rinse.

Measurement of the pH of 2HR-HNO₃ and 4HR-HNO₃ samples using broad range pH paper indicated pH values of ~2. Reaction of the nitric acid with the coupon metal likely resulted in this decrease in acidity.

Table 3. Changes in Coupon Weight and Dimension

Measurement	Coupon 1 (2 hr)	Coupon 2 (4 hr)
Initial Wt. (g)	43.3533	43.0642
Final Wt. (g)	43.2043	42.8446
Weight Loss (g)	0.1490	0.2196
Initial Diameter (mm)	26	26
Final Diameter (mm)	26	26
Diameter Decrease (mm)	0	0
Initial Thickness (mm)	10	10
Final Thickness (mm)	10	10
Thickness Decrease (mm)	0	0

2 2 Lewisite Results

Samples for Lewisite analysis from Rinse Test 2 were collected and derivatized on 10/7/09. The samples were then analyzed by GC/MS on 10/12/09. Results from the analyses are shown in Table 4. As can be seen, similar, high levels of L1 were detected in both the 2HR-HNO₃ and 4HR-HNO₃ samples. The concentration of L1 decreased for each subsequent water rinse, however, the rinse concentrations were greater than what would be expected for a 1:135 dilution, indicating that additional L1 was removed from the jar and coupon during each subsequent rinse. Extraction of the coupons and jars also resulted in the detection of L1. The four hour nitric acid rinse did not differ substantially from the two hour rinse when like samples are compared, e.g., the 4HR-DI-R2 sample had a similar L1 concentration to the 2HR-DI-R2 sample. Note that while rinse sample concentrations are expressed in µg/L, the rinsate total volume was only 136 mL. To determine the actual mass of L1 for each sample the concentration would need to be multiplied by this rinse volume. This would allow a more direct comparison of the amount of L1 in the rinse samples to the coupon and jar samples.

The distribution of L2 across the rinse, coupon and jar samples was also calculated. The L2 peak area response in each sample, taking sample preparation and dilutions into account, was calculated. The summed response was then calculated for the 2 Hr Nitric Acid Rinse and the 4 Hr Nitric Acid Rinse. The peak area response for each sample was then expressed as a percentage of the sum. As can be seen from Table 4, the majority of L2 appears to have been dissolved by the initial nitric acid rinse for both the 2 hr and 4 hr rinses, longer rinse time did not appear to increase L2 solubilization. Greater than 10% of the L2 appears to have remained associated with the TC coupon after all of the rinses were performed.

Samples 2HR-HNO₃ and 4HR-HNO₃ were stored at room temperature for 20 days. A sub-sample was collected from each of these samples on 10/27/09, diluted 10:1 with DI water and then extracted and analyzed by GC/MS to determine L1 concentration, Table 5 shows the concentration of each sample.

An additional test was performed on 11/13/09 to determine if a second 3.0 M nitric acid rinse would further reduce the concentration of L1 in the initial 3.0 M nitric acid rinse. 1.0 mL of each sample, 2HR-HNO₃ and 4HR-HNO₃, was placed in Jar 1 and Jar 2, respectively. 135 mL of 3.0 M nitric acid was added to each jar and the jar rotated for 1 hr at ~2 rpm. The sample of this second nitric acid rinse from Jar 1 was labeled 2HR-HNO₃ R2 and the sample from Jar 2 was labeled 4HR-HNO₃ R2. Sub-samples of 2HR-HNO₃ and 4HR-HNO₃ were also collected for analysis, no dilution was performed on these sub-samples prior to extraction. Results of the GC/MS analysis are shown in Table 6. Note that the concentrations of the 2HR-HNO₃ and 4HR-HNO₃ samples are about 4-fold higher compared to the previous preparation on 10/27/09. It is believed that the preparation on 10/27/09, in which 1.0 mL of sample was diluted to a final volume of 10 mL with DI water, resulted in loss of L1 due to solubility limitations of the DI water. Since the preparation of the samples on 11/13/09 used the actual sample (not a dilution) these results should more accurately reflect the actual concentration of L1 in each sample. Therefore, there has been approximately a 99.1% reduction of L1 in both sample 2HR-HNO₃ and sample 4HR-HNO₃ over time. The additional 3.0 M nitric acid rinse did not reduce the amount of L1, the concentration actually increased relative to the dilution caused by mixing 1.0 mL of sample with 135 mL of nitric acid. This may be due to additional extraction of L1 from the residual material remaining in Jar 1 and Jar 2.

Table 4 Rinse Test 2 Lewisite Results (Prepared 10/7/09)

Rinse Test	Data File	Sample ID	L1 Concentration		L2 Distribution*
2 Hr Nitric Acid Rinse	10120916 D	2HR-HNO3	12,700,000	µg/L	67%
	10120917 D	2HR-DI-R1	138,000	µg/L	13%
	10120918 D	2HR-DI-R2	17,600	µg/L	5.7%
	10120920 D	2HR-DI-R3	8,250	µg/L	2.9%
	10120926 D	L-COUPON 1	1,800	µg/coupon	11%
	10120929 D	L-JAR 1	104	µg/jar	0.5%
4 Hr Nitric Acid Rinse	10120921 D	4HR-HNO3	13,000,000	µg/L	64%
	10120922 D	4HR-DI-R1	155,000	µg/L	15%
	10120923 D	4HR-DI-R2	17,500	µg/L	3.4%
	10120924 D	4HR-DI-R3	6,390	µg/L	3.5%
	10120927 D	L-COUPON 2	849	µg/coupon	14%
	10120930 D	L-JAR 2	54.0	µg/jar	0.5%

* Percentage values for each sample do not sum to 100% due to rounding

Table 5 Rinse Test 2 Lewisite Re-Analyses (Prepared 10/27/09)

Data File	Sample ID	L1 Concentration	
10280914 D	2HR-HNO3	27,100	µg/L
10280917 D	4HR-HNO3	34,000	µg/L

Table 6 Additional Nitric Acid Rinse (Prepared 11/13/09)

Data File	Sample ID	L1 Concentration	
11160914 D	2HR-HNO3	108,000	µg/L
11160915 D	4HR-HNO3	118,000	µg/L
11160918 D	2HR-HNO3 R2	2,090	µg/L
11160920 D	4HR-HNO3 R2	2,250	µg/L

Table 7, Table 8 and Table 9 show the results of the QC samples prepared with the rinse samples and with each extraction batch. Coupon 3 (L-Coupon 3) was a clean coupon spiked with Lewisite. Recovery of L1 (59%) was similar to what was observed during the first set of rinse tests. Laboratory control samples and matrix spikes had acceptable L1 recoveries. No L1 was detected in any blanks above the detection limit. RPDs for batch duplicates and MS/MSDs were low, all less than 10%, indicating good reproducibility. On 10/7/09 the MS/MSD was prepared using sample 4HR-HNO3, because 200 fold dilutions were performed to get these samples within the instrument calibration range spike recoveries could not be calculated. Note that two sets of blanks and LCSs were prepared on 10/27/09 – one set using DI water as the matrix and the other set using 3.0 M nitric acid as the matrix. Only 3.0 M nitric acid was used for the blank and LCS on 11/13/09.

Table 7 Lewisite Sample QC (Prepared 10/7/09)

Data File	QC Sample	L1 Conc	Recovery	RPD
10120914 D	Blank	<50 µg/L		
10120915 D	LCS	6,000 µg/L	120%	
10120932 D	2HR-HNO3 DUP	11,700,000 µg/L		7.7%
10120928 D	L COUPON 3	1.5 µg/coupon	59%	

Table 8 Lewisite Sample QC (Prepared 10/27/09)

Data File	QC Sample	L1 Conc	Recovery	RPD
10280920 D	DI Blank	<50 µg/L		
10280921 D	DI LCS	4,490 µg/L	90%	
10280920 D	3.0 M Nitric Blank	<50 µg/L		
10280921 D	3.0 M Nitric LCS	5,770 µg/L	115%	
10280915 D	2HR-HNO3 DUP	29,300 µg/L		7.6%
10280918 D	4HR-HNO3 MS*	100,000 µg/L	132%	4.4%
10280919 D	4HR-HNO3 MSD*	95,900 µg/L	124%	

* Extraction batch MS/MSD

Table 9 Lewisite Sample QC (Prepared 11/13/09)

Data File	QC Sample	L1 Conc	Recovery	RPD
11160912 D	3.0 M Nitric Blank	<50 µg/L		
11160913 D	3.0 M Nitric LCS	5,380 µg/L	108%	
11160919 D	2HR HNO3 R2 DUP	1,930 µg/L		8.3%
11160921 D	4HR-HNO3 R2 MS*	8,250 µg/L	120%	1.4%
11160922 D	4HRHNO3 R2 MSD*	8,140 µg/L	118%	

* Extraction batch MS/MSD

2.3 As and Hg Results

Rinse samples for As and Hg analysis from Rinse Test 2 were collected on 10/7/09. The coupon and jar extracts were collected on 10/9/09. Samples were further prepared by microwave assisted digestion on 10/19/09 and analyzed by ICP/MS on 10/22/09. Results from the analyses are shown in Table 10 and Table 12. As can be seen, similar, high levels of As and Hg were detected in samples 2HR-HNO3 and 4HR-HNO3. The concentration of As and Hg decreased for each subsequent water rinse, however, the rinse concentrations were greater than what would be expected for a 1:135 dilution, indicating that, as with the L1, additional As and Hg was removed from the jar and coupon during each subsequent rinse. Extraction of the coupons and jars also resulted in the detection of As and Hg. The mass (mg) of As and Hg in each sample is also shown in the tables; for the rinse samples these values were determined by multiplying the sample concentration by the 136 mL rinse volume. The total As and Hg

recovered for each rinse test (2 hr Nitric Acid rinse and 4 hr Nitric Acid Rinse) was then calculated

Sample QC results are shown in Table 11 and Table 13. Neither As or Hg were detected in the preparation blank samples. For both As and Hg, laboratory control sample and matrix spike sample recoveries were acceptable and MS/MSD RPDs were low, indicating good sample preparation reproducibility. Coupon 3 was a clean coupon that was spiked with As and Hg and then extracted. The recovery of As was somewhat high (168%) and the recovery of Hg was somewhat low (49%). Both of these may be related to the low spike mass of 1.0 µg of each metal. In the case of As, the high As levels in the rinse samples could have resulted in some low-level cross-contamination leading to a high spike recovery. Alternately, there may have been some loss of Hg during the coupon extraction. These spike results relate only to the coupon extraction.

In an attempt to determine recovery of As and Hg effected by the rinsing, the total mass input of As and Hg for each rinse test was calculated. Using the mass of Lewisite liquid and Lewisite sludge added to each jar and the known metals concentration in the liquid and sludge the total mass (mg) of As and Hg could be calculated, as shown in Table 14 and Table 15. The total mass (mg) from each rinse test (2 hr Nitric Acid Rinse and 4 hr Nitric Acid Rinse) of As (Table 10) and Hg (Table 11) are also shown in Table 14 and Table 15. Both rinse tests appear to have completely recovered the As associated with the Lewisite liquid and the Lewisite sludge. However, the recovery of Hg was only about 2% for each rinse test. As most of the Hg is associated with the sludge (only ~1% of the Hg was in the Lewisite liquid), this low recovery is likely due to an inability of the 3.0 M nitric acid to dissolve the sludge material. Residual sludge remaining in the bottoms of Jar 1 and Jar 2 can be seen in Figure 8.

Table 10 Rinse Test 2 Arsenic Results

Rinse Sample ID	Arsenic Concentration		As Mass (mg)
2 HR-HNO3	4,956,000	µg/L	674
2 HR-DI-R1	167,140	µg/L	22.7
2 HR-DI-R2	44,582	µg/L	6.06
2 HR-DI-R3	27,190	µg/L	3.70
M-COUPON-1	899	µg/coupon	0.90
M-JAR-1	13,348	µg/jar	13.3
2 Hr Nitric Acid Rinse Total			721
4 HR-HNO3	4,853,300	µg/L	660
4 HR-DI-R1	179,560	µg/L	24.4
4 HR-DI-R2	44,708	µg/L	6.08
4 HR-DI-R3	23,640	µg/L	3.22
M-COUPON-2	654	µg/coupon	0.65
M-JAR-2	880	µg/jar	0.88
4 Hr Nitric Acid Rinse Total			695

Table 11 Arsenic Sample QC

QC Sample	As Conc	Recovery	RPD
Blank	< 1.0 µg/L		
LCS	20 µg/L	101%	
M-COUPON-3	67 µg/coupon	168%	
M-JAR-2 MS*	22 µg/coupon	92%	7.5%
M-JAR-2 MSD*	21 µg/coupon	85%	

* Extraction batch MS/MSD

Table 12 Rinse Test 2 Mercury Results

Rinse Sample ID	Mercury Concentration		Hg Mass (mg)
2 HR-HNO3	16,553	µg/L	2.25
2 HR-DI-R1	321	µg/L	0.044
2 HR-DI-R2	57.4	µg/L	0.008
2 HR-DI-R3	22.2	µg/L	0.003
M-COUPON-1	3.32	µg/coupon	0.003
M-JAR-1	1.10	µg/jar	0.001
2 Hr Nitric Acid Rinse Total			2.31
4 HR-HNO3	12,221	µg/L	1.66
4 HR-DI-R1	174	µg/L	0.024
4 HR-DI-R2	42.0	µg/L	0.006
4 HR-DI-R3	27.3	µg/L	0.004
M-COUPON-2	1.23	µg/coupon	0.001
M-JAR-2	0.09	µg/jar	0.00009
4 Hr Nitric Acid Rinse Total			1.70

Table 13 Mercury Sample QC

QC Sample	Hg Conc	Recovery	RPD
Blank	< 0.5 µg/L		
LCS	19 µg/L	97%	
M-COUPON-3	20 µg/coupon	49%	
M-JAR-2 MS*	21 µg/coupon	106%	0.2%
M-JAR-2 MSD*	21 µg/coupon	107%	

* Extraction batch MS/MSD

Table 14. Rinse Test 2 Arsenic Recovery

Rinse Test	D79685-L-01		D79703-S-01		Total As Added (mg)	Total Rinse Sample As (mg)	Rinse As Recovery
	Lewisite Added (g)	Lewisite As Conc. (mg/g)	Sludge Added (g)	Sludge As Conc. (mg/g)			
2 Hr Nitric Rinse	1.8610	321	0.2375	156	634	721	114%
4 Hr Nitric Rinse	1.8907	321	0.2348	156	644	695	108%

Table 15. Rinse Test 2 Mercury Recovery

Rinse Test	D79685-L-01		D79703-S-01		Total Hg Added (mg)	Total Rinse Sample Hg (mg)	Rinse Hg Recovery
	Lewisite Added (g)	Lewisite Hg Conc. (mg/g)	Sludge Added (g)	Sludge Hg Conc. (mg/g)			
2 Hr Nitric Rinse	1.8610	0.528	0.2375	428	103	2.31	2.3%
4 Hr Nitric Rinse	1.8907	0.528	0.2348	428	101	1.70	1.7%



Figure 8. Photos of residual sludge after 2 hr nitric acid rinse (left) and after 4 hr nitric acid rinse (right).